APPENDIX B

QUALITY ASSURANCE PROJECT PLAN FOR THE RCRA FACILITY INVESTIGATION AT REFINED METALS CORPORATION SITE U.S. EPA ID NUMBER IND000718130

Prepared For:

REFINED METALS CORPORATION
Beech Grove, Indiana

Prepared By:

ADVANCED GEOSERVICES CORP.
Chadds Ford, Pennsylvania

REVISION 2
March 3, 1999
98-478-01

APPENDIX B

QUALITY ASSURANCE PROJECT PLAN FOR THE RCRA FACILITY INVESTIGATION AT REFINED METALS CORPORATION SITE U.S. EPA ID NUMBER IND000718130

Prepared For:

REFINED METALS CORPORATION
Beech Grove, Indiana

Prepared By:

ADVANCED GEOSERVICES CORP.
Chadds Ford, Pennsylvania

REVISION 2
March 3, 1999
98-478-01

Quality Assurance Project Plan for the RCRA Facility Investigation at Refined Metals Corporation Site Beech Grove, Indiana U.S. EPA ID Number IND000718130 Revision 2 March 3, 1999

Prepared by: Advanced GeoServices Corp.

Prepared for: Refined Metals Corporation

Paul Stratman P.E., AGC Project Manager
Advanced GeoServices Corp.

3/3/99
Date

Denise McGuire, Quality Assurance Manager
Advanced GeoServices Corp.

Date

Douglas E. Kriscunas, Laboratory Program Manager
TriMatrix Laboratories, Inc.

Date

Matthew Love, Project Manager
Refined Metals Corporation

3/2/99
Date

Jon Adenuga, Project Manager
USEPA Region 5

Date

Brian Freeman, Permitting QA Coordinator
USEPA RCRA Enforcement

Date

TABLE OF CONTENTS

PAGE NO.

TABLE OF CONTENTS
(Continued)

PAGE NO.

TABLE OF CONTENTS
(Continued)

PAGE NO.

TABLE OF CONTENTS
(Continued)

PAGE NO.

TABLE OF CONTENTS
(Continued)

PAGE NO.

TABLE OF CONTENTS
(Continued)

PAGE NO.

TABLE OF CONTENTS
(Continued)

PAGE NO.

TABLE OF CONTENTS
(Continued)

PAGE NO.

LIST OF TABLES

TABLE

LIST OF FIGURES

FIGURE

LIST OF ATTACHMENTS

ATTACHMENT

List of Persons Who Have Received This QAPP

AGC
Exide Corporation
Swidler & Berlin
Indiana DEM
U.S. Department of Justice
U.S. EPA Region 5

1.0 PROJECT DESCRIPTION

1.1 INTRODUCTION

On behalf of Refined Metals Corporation (RMC) Advanced GeoServices Corp. (AGC) has prepared this Quality Assurance Project Plan (QAPP) for the proposed RFI activities at the RMC Facility in Beech Grove, Indiana (Site). A Project Management Plan, QAPP (including Data Management Plan), Health and Safety Plan, and Community Relations Plan have been appended to the RCRA Facility Investigation (RFI) Work Plan, dated March 3, 1999.

1.1.1 Overall Project Objectives and Decision Statements

The objectives of this RFI are to determine the nature and extent of contamination at or migrating off-site from the facility and to gather sufficient information to quantify risk to human health (baseline risk assessment) and ecological receptors (preliminary ecological risk assessment) in the event that environmental contamination is determined to be present.

Overall objectives of the data collection will be as follows:

- Verify and further define the presence, magnitude, extent, and mobility of hazardous waste and hazardous waste constituents on and beneath the former site area and adjacent off-site areas that may have originated from the RCRA permitted hazardous waste or solid waste management units at the Site.
- Collect sufficient data to quantify risks to human health and the environment. Laboratory data will eventually be compared to established human health and ecological target decision levels. The target decision levels are summarized in Table 1-1.

- Collect sufficient data for groundwater, soil, sediment and building interior dust to support a Corrective Measures Study to evaluate and develop alternatives and recommend a final corrective measure

The on-site and off-site sampling program outlined in the RFI Work Plan and the principles and procedures set forth in this QAPP are designed to ensure that data are of sufficient quality to perform comparisons with target decision levels and to quantify risk to human health and ecological receptors. The Decision Statement for this investigation is as follows: Identify the nature and extent of RCRA metals and select volatile organic compounds (VOCs) in on-site and off-site soil, on-site sediment, groundwater and building interior dust that present unacceptable risks, which would therefore warrant remedial action.

1.1.2 Project Status/Phase

AGC will utilize an integrated and phased approach for the RFI at the RMC Site. During the RFI, data collection will be conducted in phases, with the results of the human health baseline risk assessment and preliminary ecological risk assessment being determining factors in decisions regarding the necessity for additional phases of investigation.

The Phase I field investigation will include the following activities:

- Surface and subsurface soil sampling for verification and Site characterization both on-site and off-site;
- Phased groundwater investigation (evaluation of current network of wells and low-flow sampling);
- On-site sediment sampling; and
- Building interior dust and subsurface soil sampling.

Target analytes for field and laboratory analysis are discussed in Section 1.4 of this QAPP.

Data from the Phase I investigation will be evaluated to determine whether a Phase II investigation is necessary. If Phase I data indicate that sufficient Site characterization information has been collected, and the data quality objectives have been met, RMC will proceed with the baseline human health risk assessment and the preliminary ecological risk assessment for the Site, if applicable (i.e., if target levels for human health or ecological receptors are exceeded). An RFI Report presenting the Phase I data and recommendations of the risk assessments will be prepared and submitted to the USEPA. After a review of the RFI Report, the need for implementing a Phase II investigation will be evaluated in light of the data requirements for the feasibility study. The rationale and scope of any Phase II investigation will be discussed with and approved by the USEPA prior to implementation.

1.1.3 QAPP Preparation Guidelines

This QAPP has been prepared in accordance with the Region 5 QAPP policy as presented in U.S. EPA RCRA QAPP Instructions, dated April 1998. Originally, the QAPP was performed using Region 5 QAPP policy dated May 1993 that was provided with the Order and was submitted to the client for review in September 1999, upon which the new QAPP policy (April 1998) was made available.

1.2 SITE/FACILITY DESCRIPTION

1.2.1 Location

The Refined Metals Corporation Site is located at 3700 Arlington Avenue, Beech Grove, Marion County, Indiana, in a zone of mixed land uses. The physiographic setting is described in Section 3.1 of the RFI Work Plan.

1.2.2 Facility/Site Size and Borders

The Site encompasses approximately 24 acres, and is bordered by a railroad spur on the north, a Firestone facility that manufactures roofing materials on the east (across Arlington Avenue), and a mix of vacant and industrial properties to the south. A Citizens Gas Storage facility and pipeline are located northwest of the Site, and a railroad yard and repair facility and Conrail and Amtrak are located beyond Citizens Gas toward the northwest.

1.2.3 Natural and Manmade Features

Section 3.0 of the RFI Work Plan discusses natural features on and surrounding the Site, and Section 2.0 of the RFI Work Plan describes the manmade features of the facility.

1.2.4 Topography

See Section 3.2 and Figure 3-1 of the RFI Work Plan for information concerning the Site topography and drainage.

1.2.5 Local Geology and Hydrogeology

See Sections 3.4 and 3.5 of the RFI Work Plan for information concerning the regional and local geology and hydrogeology of the Site.

1.2.6 Surrounding Land Use

See Section 3.3 of the RFI Work Plan for a discussion on surrounding land use.

1.2.7 Ecological Communities and Habitats

See Section 3.6 of the RFI Work Plan for a discussion of the ecological setting as determined through a site visit.

1.3 SITE/FACILITY HISTORY

1.3.1 General History

Refined Metals Corporation was engaged in recycling lead batteries and other lead wastes. There are currently no manufacturing operations at the facility. The plant was constructed in 1968 as a secondary lead smelter. In 1984, a battery breaker operation was installed. From April 14, 1995, through December 31, 1995, operations were reduced to enriching and casting lead ingots from off-specification lead products.

The facility was constructed to recycle lead batteries and other lead wastes. Automotive batteries constituted 90 percent of the materials recycled, and the remainder was waste material from battery manufacturers and other lead scrap. During operation, the batteries were temporarily stored in

trailers or on pallets in a paved storage yard. The batteries were then fed into the battery crusher, where the tops of the batteries were sawed off and the sulfuric acid was drained into a stainless steel tank that drained to the wastewater treatment system. The battery casings and their contents were tumbled and crushed. Lead plates and other lead parts were separated and transported to the materials storage building to be later placed in the furnace. The battery casings were shredded and separated into plastic and rubber in a flotation tank. The plastic was blown into a trailer for sale to be sold to an off-site recycler. Rubber was stored and then fed into the blast furnace.

Before 1984, materials were stored on-site with minimal spill or runoff control. Storm water runoff from the storage piles and work areas flowed to the storage pond and evaporated; some runoff flowed off site to the north drainage ditch. Once the battery crusher was installed in 1984, a batch neutralization system was installed to treat acidic wastewater from the battery crushing and flotation systems. The wastewater was neutralized before discharging to Beech Grove Municipal Sanitary Sewer system. Since 1988, all stormwater has been contained and routed to the wastewater treatment system.

Reportedly, underground storage tanks (USTs) were never used at the Site. Three above ground storage tanks (ASTs) - two 10,000-gallon (ASTs) and one 20,000-gallon AST - were used to store diesel fuel for company trucks. The tanks were reportedly cleaned out in 1985 and are out of service. The three tanks are enclosed by a spill containment wall which was reportedly constructed before 1980. A 500-gallon AST and a 750-gallon AST were used for diesel fuel and gasoline, respectively, to fuel on-site vehicles. The 750-gallon gasoline tank is enclosed within a spill containment wall and pad. Propane, which is used to power forklifts, is stored in a 2,000-gallon tank.

A leak in a valve of one of the out-of-service diesel tanks occurred around 1983, resulting in a spill outside of the containment wall. A portion of the spill flowed along the drainage ditch located north of the refining area. The contaminated soil was excavated and the tanks were emptied. Although documentation of the spill is not available, the soil cleanup was reportedly conducted under state supervision.

1.3.2 Past Data Collection Activities

Elevated concentrations of lead have been detected in surface and subsurface (less than four feet below ground surface) soils at the Site. Low levels of lead and arsenic have been detected in unfiltered groundwater samples (and on one occasion a filtered sample that is believed to be a discrepancy) collected from the Site. See Section 5.0 of the RFI Work Plan for more information concerning previous sampling efforts and analytical results for the Site. Prior data has been used as a screening tool to assist RMC in developing the proposed RFI sampling program; however, prior data will not be used to determine risk to human health or ecological receptors.

1.3.3 Current Status

Since 1996, no production has taken place and the facility has been inactive.

1.4 PROJECT OBJECTIVES AND INTENDED DATA USAGES

For this project, it will be necessary to gather sufficient information to evaluate the nature and extent of releases from solid waste management units, and also to determine whether unreasonable risks to human and ecological receptors are associated with the areas.

The RFI activities will include:

- Evaluation of the existing monitoring well network;
- Low-flow groundwater sampling using the existing network;
- Surface and subsurface soil sampling, on and off-site;
- Dust and subsurface soil sampling inside of the buildings; and
- Sediment sampling of the surface impoundment and former drainage channels on the facility.

Data collection activities will specifically address the following concerns:

- The nature and extent of on and off-site soil (surface and subsurface), on-site sediment, groundwater, and interior dust contamination;
- The impact of potential soil, sediment, and groundwater contamination on human health; and
- The impact of potential soil and sediment contamination on ecological receptors.

Parameters listed in Table 1-1 are the proposed critical measurement parameters for this project. Section 5.0 of the RFI Work Plan describes the details of the surface and subsurface soil, groundwater, sediment, and interior dust sampling activities.

AGC risk assessment staff have reviewed the media sampling programs as proposed in the RFI Work Plan and this QAPP to ensure that data collection activities will be in accordance with USEPA guidance for data quality objectives (USEPA 1987a,b). The following sequential steps will be performed by AGC risk assessment staff proceeding the collection of validated data (see Section 9.0) from RFI sampling:

1. On and off-site soil (including subsurface soil underneath buildings), sediment, and groundwater data will be compared to the target decision levels for human health. The human health decision levels are derived from the following sources:

   - Soil - USEPA Region 9 Preliminary Remediation Goals (PRGs) for Industrial Soil; and
   - Groundwater - USEPA Region 9 PRGs for Tap Water.

   If the maximum constituent concentration for a particular medium exceeds the decision level, all data for that medium will be used for the human health risk assessment.

2. Soil and sediment data collected from the three major habitats identified at the Site will be compared to the target decision levels for ecological receptors. The ecological decision levels are derived from the following sources:

   - Soil - Region 5 Ecological Data Quality Levels; and
   - Sediment - Region 5 Ecological Data Quality Levels.

   If the maximum constituent concentration for a particular medium/habitat exceeds the decision level, all data for that medium/habitat will be used for the preliminary ecological risk assessment.

3. For those media/habitats that require risk assessment, human and/or ecological receptors will be identified. Human receptors will be based on sensitive subpopulations associated with current (i.e., industrial) and potential future land use. The selection of ecological receptors will be based on Habitat Suitability Indices (US Fish and Wildlife).

4. The reasonable maximum exposure concentration for each medium/habitat will be estimated by the upper 95% confidence limit on the mean of the medium/habitat data.

5. Risk assessments will be performed to determine cumulative risk using the reasonable maximum exposure concentration for each medium/habitat in accordance with the following primary guidance documents:

   - Human Health Risk Assessment - USEPA. 1989. Risk Assessment Guidance for Superfund Volume 1: Human Health Evaluation Manual (Part A); and

- Preliminary Ecological Risk Assessment - USEPA 1997. Ecological Risk Assessment Guidance for Superfund: Process for Designing and Conducting Ecological Risk Assessments, Interim Final.

The decision rule associated with sampling activities is that if any constituent from Table 1-1 is identified above the human health or ecological target levels (where applicable), then all validated data collected for a particular medium (i.e., soil, groundwater, sediment) will be used in a baseline human health risk assessment and/or preliminary ecological risk assessment. It should be noted that previous data collected at the Site will not be used for assessing risk; thus, data acquisition requirements are not needed and not discussed in this QAPP. If no constituents in Table 1-1 are detected above target levels, then the baseline human health risk assessment and preliminary ecological risk assessment will be performed using one-half the reporting limit values for the constituents in Table 1-1 which analytical sensitivity was inadequate. If the risks are deemed "not unreasonable," then the Site may qualify for "No Further Action."

1.4.1 Project Target Parameters

Based on past sampling data and as stated in the Consent Decree, the primary constituents of concern at the Site are lead and cadmium. As discussed and agreed with USEPA, soil and sediment samples will be analyzed for arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver (RCRA metals). The short list of regulated constituents for diesel fuel will be analyzed in select soil samples (see Section 5.0 of RFI Work Plan). Interior dust samples will be analyzed for lead and cadmium. Groundwater samples will be analyzed for RCRA metals and antimony. As agreed with the EPA groundwater samples during the first event will also be analyzed for certain VOCs, including tetrachloroethylene (PCE), 1,1,1-trichloroethane (TCA), benzene, toluene, and ethylbenzene. If the VOCs are not detected they will not be analyzed in subsequent groundwater sampling events. Sampling parameters and quantitation limits are listed on Table 1-1.

1.4.2 Field Parameters

Low-flow sampling indicator parameters such as temperature, pH, redox potential, dissolved oxygen, turbidity, and specific conductance will be monitored in the field during well purging (for monitoring wells) and at the time of sample acquisition to assure that the well has been adequately purged and that the groundwater is a representative sample from the aquifer. No field parameters will be collected during soil, sediment or dust collection.

1.5 SAMPLING LOCATIONS

1.5.1 Rationale of Selected Sampling Locations

Maps showing the proposed soil, sediment, and dust/subsurface sampling locations are provided on Figures 5-1, 5-2 and 5-3 of the RFI Work Plan. These locations are proposed and depending on the nature of encountered field conditions (i.e., if a tree is present, or soil is inpenetratable), sampling locations may be changed by a few feet. The On-Site Principle Investigator will be responsible for making such decisions. Locations of the monitoring wells to be sampled are indicated on Figure 1-2 of the RFI Work Plan. The rationale for the selected sampling locations are fully described in Sections 5.2, 5.3, 5.4 and 5.5 of the RFI Work Plan.

1.6 PROJECT SCHEDULE

1.6.1 Anticipated Date of Project Mobilization

It is anticipated that RFI activities will begin immediately after the approval of the RFI Work Plan.

1.6.2 Task Bar Chart and Associated Time Frames

The estimated Project Schedule shown on Figure A-2 of the RFI Work Plan indicates the RFI activities will be conducted in a sequence of progressive work tasks. The RFI Work Plan and attachments delivery, review, and approval by USEPA and IDEM comprise the first task.

TABLE 1-1
REFINED METALS SITE PROJECT ANALYTE LIST

Constituent	Matrix	Human Health[1] Data Quality Level[1]	Ecological Data Quality Level[3]	Reporting Limit
Antimony	aqueous[1]	15 ug/l	Not Applicable	10 ug/l
Antimony	soil[2]	7.5e[2] mg/kg	0.142 mg/kg	4.0 mg/kg
Antimony	sediment	Not Applicable	Not Applicable	4.0 mg/kg
Arsenic (cancer endpoint)	aqueous	0.045 ug/l*	Non Applicable	1 ug/l
Arsenic (cancer endpoint)	soil	3 mg/kg	5.7 mg/kg	4.0 mg/kg
Arsenic	sediment	Not applicable	5.9 mg/kg	4.0 mg/kg
Barium	aqueous	2,600 µg/l	Not Applicable	10 µg/l
Barium	soil	1e[5] mg/kg	Not Applicable	0.4 mg/kg
Barium	sediment	Not Applicable	Not Available	0.4 mg/kg
Cadmium	aqueous	18 ug/l	Not Applicable	0.2 ug/l
Cadmium	soil	9.3e[2] mg/kg	0.181 mg/kg	0.5 mg/kg
Cadmium	sediment	Not Applicable	0.596 mg/kg	0.5 mg/kg
Cadmium	dust	Not Applicable	Not Applicable	0.5 mg/kg
Chromium	aqueous	Not Applicable	Not Applicable	
Chromium	soil	4.5e[2] mg/kg	0.4 mg/kg	
Chromium	sediment	Not Applicable	26 mg/kg	
Lead	aqueous	4 ug/l	Non Applicable	1 ug/l
Lead	soil	1e[3] mg/kg	0.450 mg/kg	0.7 mg/kg
Lead	sediment	Not Applicable	31 mg/kg	0.7 mg/kg
Lead	dust	Not Applicable	Not Applicable	0.7 mg/kg
Selenium	aqueous	180 µg/l	Not Applicable	2.0 µg/l
Selenium	soil	9.4e[3] mg/kg	0.28 mg/kg	4.0 mg/kg
Selenium	sediment	Not Applicable	Not Available	4.0 mg/kg
Silver	aqueous	180 µg/l	Not Applicable	0.2 µg/l

TABLE 1-1
REFINED METALS SITE PROJECT ANALYTE LIST (continued)

Constituent	Matrix	Human Health[1] Data Quality Level[1]	Ecological Data Quality Level[3]	Reporting Limit
Silver	soil	9.4e[3] mg/kg	4.04 mg/kg	0.2 mg/kg
Silver	sediment	Not Applicable	0.5 mg/kg	0.2 mg/kg
Mercury	aqueous	11 µ/l	Not Applicable	0.2 µg/l
Mercury	soil	5.6e[2] mg/kg	0.0079 mg/kg	0.1 mg/kg
Mercury	sediment	Not Applicable	0.174 mg/kg	0.1 mg/kg
Benzene	aqueous	0.39 µg/l	Not Applicable	
Benzene	soil	1.4e°	.255 mg/kg	0.05 mg/kg
Cumene	soil	5.2e°	Not Available	0.05 mg/kg
Ethylbenzene	aqueous	1,300 µg/l	Not Applicable	1 µg/l
Ethylbenzene	soil	230 mg/kg	Not Applicable	0.05 mg/kg
Toluene	aqueous	720 µg/l	253 µg/l	1 µg/l
Toluene	soil	5.2e[2] mg/kg	5.45 µg/kg	0.05 mg/kg
Tetrachloroethene	aqueous	1.1 µg/l	Not Applicable	1 µg/l
1,1,1-Trochloroethane	aqueous	790 µg/l	Not Applicable	1 µg/l
Napthalene	soil	1.9e[2] mg/kg	0.99 mg/kg	330 µg/kg
Fluorene	soil	2.2e[4] mg/kg	122 mg/kg	330 µg/kg
Phenanthrene	soil	Not Applicable	45,700 µg/kg	330 µg/kg

* - For these parameters, analytical sensitivity is inadequate to meet target decision levels. Therefore, for risk assessment purposes, non-detect data shall be considered as equal to one-half the reporting limit.

1. Region 9 Preliminary Remediation Goals (PRGs), 1998.
2. Soil values are represented by industrial soil PRGs.
3. Region 5 Ecological Data Quality Levels.

Note: The maximum concentration for each medium and constituent will be compared to the target decision levels to determine the need for human health and ecological risk assessments.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

2.1 PROJECT ORGANIZATION CHART

The Refined Metals Corporation has selected Advanced GeoServices Corp., Chadds Ford, Pennsylvania to be responsible for coordinating sampling and analysis activities and validating data received from the laboratory. TriMatrix Laboratories, Grand Rapids, Michigan, will conduct the chemical analyses of the samples. This laboratory possesses all credentials to do this work; qualifications and standard operating procedures are provided as Attachments A and B to this QAPP.

While all personnel involved in the investigation and in the generation of data are implicitly a part of the overall project and quality assurance program, certain individuals have specific responsibilities. The key individuals who are responsible for the overall coordination of efforts to be conducted, as well as the collection, validation and interpretation of the data generated during this project, are identified in the following sections. Lines of authority specific to this investigation are presented in Figure 2-1 (Project Organization Chart). The figure includes all individuals discussed below.

2.2 MANAGEMENT RESPONSIBILITIES

2.2.1 USEPA Region 5

Remedial Project Manager - Jon Adenuga

Responsibilities of the EPA Remedial Project Manager include:

- Overseeing implementation of the administrative order;

- Providing technical review and approval of all plans and data submitted as part of this investigation; and
- Coordinating site monitoring activities with RMC Project Manager

2.2.2 Refined Metals Corporation

Project Manager - Matthew Love

Responsibilities of RMC Project Manager include:

- Providing historical information regarding facility operations and processes.
- Preparing and submitting monthly updates on project progresses and other relevant information as required by the Consent Decree.
- Overseeing and coordinating all project activities on behalf of RMC.
- Reviewing and approving contract related issues, including scope of work, and approving invoices for payment.
- Reviewing and commenting on technical reports.
- Representing RMS at meetings with EPA and IDEM.
- Approving changes in the scope and direction of investigations and other technical issues.

2.2.3 Advanced GeoServices Corp.

Project Manager - Paul G. Stratman, P.E.

Responsibilities of the AGC Project Manager include:

- Managing and coordinating site monitoring;

- Reviewing information obtained during the RFI;
- Initiating any RFI Work Plan or QAPP modifications;
- Providing in-house technical support for evaluating and organizing field data; and
- Providing input to the Task Managers on technical direction.

Task Managers - Edie M. Gair, P.G. and Stephen W. Kirschner, P.E.

Responsibilities of the AGC Task Managers include:

- Managing and coordinating the tasks of the Principle Investigator and technical staff personnel;
- Reviewing information obtained during the RFI; and
- Preparation of baseline plans and for the performance of work elements that comprise this project.

2.3 QUALITY ASSURANCE RESPONSIBILITIES

2.3.1 Advanced GeoServices Corp.

Quality Assurance (QA) Manager - Denise McGuire

Responsibilities of the AGC QA Manager include:

- Conducting at a minimum one field performance audit;
- Performing data validation and assessment of the analytical data generated during the RFI;
- Communicating analytical deficiencies found during data validation to the Project and Tasks Managers to initiate corrective action;
- Preparing data validation reports and tabulation of analytical data; and

- Communicating with the laboratory for data deliverables and any problems with the data reported.

In addition, AGC Quality Assurance Scientists will be utilized to review chain-of custodies, validate data, construct data summary tables, and perform data entry. The QA Scientists will report to the QA Manager.

2.4 LABORATORY RESPONSIBILITIES

2.4.1 TriMatrix Laboratories, Inc.

Laboratory Operations Manager - Douglas E. Kriscunas

The Laboratory Operations Manager's responsibilities include:

- Liaison with sampling firm's Project Manager, Quality Assurance Manager, and laboratory technical staff;
- Production and efficiency of all departments including QA/QC;
- Recommendations of appropriate corrective action procedures to the QA Manager;
- Identification and supervision of appropriate and necessary support personnel; and
- Oversees final analytical results.

Laboratory Program Manager - Jennifer Rice

The responsibilities of the Laboratory Program Manager include:

- Coordinates laboratory analyses;
- Supervises in-house chain-of-custody;
- Oversees data review and data assessment;
- Oversees preparation of analytical reports; and
- Approves final analytical reports prior to submittal to the Client.

Laboratory Quality Assurance Supervisor (LQAS) - Rick D. Wilburn

Responsibilities of the LQAS include:

- Oversees QA/QC documentation;
- Inspecting and verifying laboratory QA/QC records and results;
- Implementing all laboratory QA/QC procedures contained in the QAPP;
- Overseeing corrective actions as required; and
- Conducting internal system and performance audits and inspection of analytical procedures.

Laboratory Sample Custodian - Keith Banchoff

The Sample Custodian's responsibilities include:

- Providing sample bottles;
- Receiving and inspecting the incoming sample bottles;
- Recording the condition of the incoming sample containers;
- Verifying chain-of-custody and it's correctness;
- Notifying Laboratory Program Manager of sample receipt and inspection;
- Assigning an unique identification number and customer number and enters each into the sample receiving log; and
- Controlling and monitoring access/storage of samples.

Laboratory Technical Staff

The technical staff will be responsible for sample analysis and identification of corrective actions. The staff will report directly to the Laboratory Program Manager.

2.5 FIELD RESPONSIBILITIES

2.5.1 Advanced GeoServices, Corp.

On-Site Principle Investigator (PI) - Eric Stanke

The PI's responsibilities include:

- Providing full time field representation during field data collection activities;
- Collecting and reporting raw data;
- Overseeing any site contractors and other field personnel to ensure adherence to the RFI Work Plan and QAPP; and

Ensuring the appropriate QC samples are collected.

Field Technical Staff

The technical staff for this project will be drawn from AGC's pool of corporate resources. The technical staff will be utilized to gather and analyze data, and to prepare various task reports and support materials. All of the designated technical team members are experienced professionals who possess the degree of specialization and technical competence required to effectively and efficiently perform the required work.

2.6 SPECIAL TRAINING REQUIREMENTS AND CERTIFICATION

2.6.1 Training

All personnel performing on-site activities are 40 hour OSHA 1910.120 trained. These individuals include:

Paul Stratman, P. E
Edie M. Gair, P.G.
Stephen W. Kirschner, P. E.
Denise McGuire
Eric Stanke
AGC Field Technical Staff

2.6.2 Certification

Certifications required for implementing this plan have already been attained for the individuals listed above. All necessary OSHA certification documentation will be taken to the Site during field activities and available for review.

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

Data Quality Objectives (DQOs) are quantitative and qualitative statements specifying the quality of the environmental data required to support the decision making process. Separate DQOs are designed for field sampling and laboratory analysis so that clear distinctions between any problems found in the system can be isolated with respect to cause. Conversely, the DQOs are also designed to provide an indication of the variability of the overall system. The overall quality assurance objective is to keep the total uncertainty within an acceptable range that will not hinder the intended use of the data and to provide results which are legally defensible in a court of law. To achieve this, specific data requirements such as detection limits, criteria for precision and accuracy, sample representativeness, data comparability and data completeness (PARCC) are specified below. The DQOs for the RMC Site are presented in Tables 3-1 and 3-2.

3.1 PRECISION

3.1.1 Definition

Precision is a measure of the degree to which two or more measurements are in agreement.

3.1.2 Field Precision Objectives

Field precision is assessed through the collection and measurement of field duplicates at a rate of 1 duplicate per 10 analytical samples. The total number of duplicates for this project is found in Table 3-3 of this QAPP.

3.1.3 Laboratory Precision Objectives

Precision in the laboratory is assessed through the calculation of relative percent differences (RPD) and relative standard deviations (RSD) for three or more replicate samples. The equations to be used for precision in this project can be found in Section 12.1 of this QAPP. Precision control limits are provided in Table 3-2.

For inorganic analyses, laboratory precision shall be assessed through the analysis of a sample/sample duplicate pair and field duplicate pairs. For organic analyses, laboratory precision shall be assessed through the analysis of matrix spike/matrix spike duplicate (MS/MSD) and field duplicate samples. All parameters of concern listed in Table 1-1 of this QAPP are included in method spiking solutions for MS and MS/MSD analyses.

3.2 ACCURACY

3.2.1 Definition

Accuracy is defined as the degree of agreement of a measurement or average of measurements with an accepted reference value. Accuracy measures the bias in a measurement system which may result from sampling or analytical error. Sources of error that may contribute to poor accuracy are:

- laboratory error;
- sampling inconsistency;
- field and/or laboratory contamination;
- handling;
- matrix interference; and
- preservation.

3.2.2 Field Accuracy Objectives

Accuracy in the field is assessed through the use of field and trip blanks and through the adherence to all sample handling, preservation and holding times.

3.2.3 Laboratory Accuracy Objectives

Laboratory accuracy is assessed through the analysis of MS/MSD, standard reference materials (SRM), laboratory control samples (LCS) and surrogate compounds, and the determination of percent recoveries. Accuracy in laboratory methods and procedures will be evaluated by use of calibration and calibration verification procedures, and instrument performance solutions at the frequency specified in the USEPA "Test Methods for Evaluating Solid Waste Physical/Chemical Methods", November 1986, SW-846 3rd edition (SW-846) Update III. The equation to be used for accuracy in this project can be found in Section 12.2 of this QAPP. Accuracy control limits are given in Table 3-2. All parameters of concern included in Table 1-1 of this QAPP are included in method spiking solutions for the LCS and MS/MSD samples. Also, in the case of sampling for VOCs in soil, use of the use of the Encore sampler will ensure data that is both accurate and representative of on-site conditions.

3.3 DATA COMPLETENESS

3.3.1 Definition

Completeness is defined as the percentage of data that is judged to be valid to achieve the objectives of the investigation compared to the total amount of data.

3.3.2 Field Completeness Objectives

Field completeness is a measure of the amount of valid measurements taken in the project. The equation for completeness is presented in Section 12.3 of this QAPP. The field completeness objective for this project will be greater than 90 percent.

3.3.3 Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of valid measurements obtained from all measurements taken in the project. The equation for completeness is presented in Section 12.3 of this QAPP. The laboratory completeness objective for this project, with respect to critical measurement parameters identified in Table 1-1, will be greater than 90 percent.

3.4 DATA REPRESENTATIVENESS

3.4.1 Definition

Representativeness expresses the degree to which sample data represent the characteristics of the environment from which they are collected. Samples that are considered representative are properly collected to accurately characterize the contamination at a sample location.

3.4.2 Measures to Ensure Representativeness of Field Data

Representativeness is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the RFI Work Plan is followed and that proper sampling techniques are used. Representativeness will be measured by using the field methods (e.g., sampling, handling, and preserving) in accordance with NEIC Policies and Procedures Manual and analytical methods in accordance with SW-846 methodologies.

3.4.3 Measures to Ensure Representatives of Laboratory Data

Representatives in the laboratory is ensured by using the proper analytical procedures, appropriate methods, meeting sample holding times and analyzing and assessing field duplicate samples. The sampling network was designed to provide data representative of facility conditions. During development of this network, consideration was given to past waste disposal practices, the physical setting, and constraints inherent to the RCRA program. The rationale of the sampling network is discussed in Section 5.0 of the RFI Work Plan.

3.5 DECISION RULES

3.5.1 Definition

A Decision Rule is a statement which allows for a course of action or non-action to be taken, based on assumptions made to draw out and test its logical or empirical consequences.

3.5.2 Decision Rule Objectives

The decision rule objectives for this investigation address the definition of statistical parameter(s) characterizing the population, identification of action levels, and development of if/then statements defining conditions that would cause the decision maker to chose among alterative actions. The decision rule associated with this investigation is that if any of the critical measurement parameters listed in Table 1-1 are identified above human health or ecological target levels in any of the monitoring wells, sediments, or soils, then the data will be used to define the extent of contamination or map the plume boundaries and all data generated will be subjected to a baseline human health risk assessment and preliminary ecological risk assessment. The decision rule will be applied to validated data obtained from RFI sampling activities with the following conditions:

- Sampling of the groundwater will not be performed until specific field parameters (i.e. redox potential, pH, specific conductance and dissolved oxygen) stabilize.

- In order to determine whether the existing monitoring well network is sufficient to detect a release to groundwater, two quarterly groundwater sampling events will be conducted. Groundwater shall be analyzed at a fixed laboratory for parameters identified in Table 1-1 site-related constituents in soil, grab samples will be taken at two depth intervals within the arbitrary grid shown on Figure 5-1 of the RFI Work Plan and from soil piles on the Site.

- Interior sampling will be performed to characterize the floor material and determine the extent of site-related constituent contamination beneath the Material Storage Building.

- Composite sediment samples will be collected in the former drainage channels and lined lagoon to address depositional environments at the Site.

The decision rule will be used following the validation of all RFI Phase I data, and the requirements for a baseline human health assessment and preliminary ecological risk assessment will be determined at that time.

3.6 COMPARABILITY

3.6.1 Definition

Comparability expresses the confidence with which one data set can be compared with another data set from a different phase or from a different program.

3.6.2 Measures to Ensure Comparability of Field Data

Comparability is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the RFI Work Plan is followed and that proper sampling techniques are used.

3.6.3 Measures to Ensure Comparability of Laboratory Data

Comparability will be accomplished by ensuring that proper sample collection techniques will be utilized and through the use of standardized and approved methods of analysis.

3.7 LEVEL OF QUALITY CONTROL EFFORT

PARCC parameters will be monitored through the submission and analyses of various types of field and laboratory QC samples. These will include appropriate field blanks, equipment blanks, laboratory method blanks, field duplicates or replicates, matrix spikes, matrix spike duplicates, instrument performance solutions, and a careful examination of all calibration and check standards. Specifically:

- Field blanks and equipment blank consisting of distilled water will be submitted to the laboratory to provide the means to assess the quality of the data resulting from the field sampling program.
- Field blank samples are analyzed to check the procedural contamination at the facility which may cause sample contamination.
- Equipment blank samples are analyzed to check the decontamination procedural for field equipment which may attribute to cross contamination.
- Method blank samples are generated within the laboratory and used to assess contamination resulting from laboratory practices.
- Duplicate samples are analyzed to check for sampling and analytical reproducibility.
- MS/MSDs are performed to provide information about the effect of the sample matrix on the digestion and measurement methodology. MS/MSD pairs also check the analytical reproducibility.
- Instrument performance solutions, calibration and check standards are analyzed to assess the capability of the laboratory to perform the specific methods.

The frequency by which the field and laboratory QC samples will be prepared and submitted is specified in Section 8.0 of this QAPP. Table 3-3 summarizes the type and frequency of QC samples to be performed during this investigation. Sampling procedures for blanks and field duplicates are provided in Section 8.1.1 and 8.1.2. Quantitation limits for the critical measurement parameters are provided in Table 3-4.

TABLE 3-1
REFINED METALS SITE
DATA QUALITY OBJECTIVES

DQO Parameter	Metals	Indicator Parameters[a]
Accuracy	Table 3-2	Table 3-2
Precision	Table 3-2	Table 3-2
Completeness	90%	100%
Comparability	Based on precision, accuracy and media comparison	Based on precision, accuracy and media comparison

Notes:

a. Indicator parameters include: specific conductance, temperature, dissolved oxygen, redox potential and pH.

TABLE 3-2
REFINED METALS SITE
ACCURACY AND PRECISION DATA QUALITY
OBJECTIVES FOR METALS AND FIELD PARAMETERS

Matrix	Audit	Analytes	Control Limits[1]
Aqueous	<u>Accuracy</u>		
	Laboratory Blank	Metals Volatile Organic Compounds	<±LOQ
	Field/Equipment Blank	Metals Volatile Organic Compounds	<±LOQ
	Matrix Spike Recovery (MS/MSD)	Metals	75-125% unless the sample concentration exceeds the spike added concentration by a factor of 4 or more
	Matrix Spike/Surrogate Spike	Volatile Organic Compounds	As specified in Laboratory SOP
	Laboratory Control Sample	Metals	80-120%
	Laboratory Control Sample	Volatile Organic Compounds	As specified in Laboratory SOP
Aqueous	<u>Precision</u>		
	Matrix Spike Duplicate/Matrix Spike Duplicate	Metals	<20% RPD for results >5 x LOQ <±LOQ for results <5 x QL

TABLE 3-2
REFINED METALS SITE
ACCURACY AND PRECISION DATA QUALITY
OBJECTIVES FOR METALS AND FIELD PARAMETERS
(Continued)

Matrix	Audit	Analytes	Control Limits[1]
		Volatile Organic Compound	As specified in Laboratory SOP
	Field Duplicate	Metals	<20% RPD for results >5 x LOQ <±LOQ for results <5 x QL
		Volatile Organic Compound	<20%RPD
Soil/Sediment/ Dust	Accuracy		
	Laboratory Blank	Metals Volatile Organic Compounds Semivolatile Organic Compounds	<LOQ
	Field/Equipment Blank	Metals Volatile Organic Compounds Semivolatile Organic Compounds	<LOQ
	Matrix Spike Recovery (MS/MSD)	Metals	75-125% unless the sample concentration exceeds the spike added concentration by a factor of 4 or more
	Matrix Spike/Surrogate Spike	Volatile Organic Compounds	As specified in Laboratory SOP

TABLE 3-2
REFINED METALS SITE
ACCURACY AND PRECISION DATA QUALITY
OBJECTIVES FOR METALS AND FIELD PARAMETERS
(Continued)

Matrix	Audit	Analytes	Control Limits[1]
		Semivolatile Organic Compounds	As specified in Laboratory SOP
	Laboratory Control Sample	Metals	80-120%
	Laboratory Control Sample		
		Volatile Organic Compounds	As specified in Laboratory SOP
		Semivolatile Organic Compounds	As specified in Laboratory SOP
Soil/Sediment/ Dust	<u>Precision</u>		
	Matrix Spike Duplicate	Metals	<20% RPD for results >5 x LOQ <±LOQ for results <5 x QL
		Volatile Organic Compounds	
		Semivolatile Organic Compounds	
	Field Duplicate	Metals	<40% RPD for results >5 x LOQ <±2xLOQ for results <5 x QL
		Volatile Organic Compounds	<30%RPD
		Semivolatile Organic Compounds	<40%RPD

TABLE 3-2
REFINED METALS SITE
ACCURACY AND PRECISION DATA QUALITY
OBJECTIVES FOR METALS AND FIELD PARAMETERS
(Continued)

Matrix	Audit	Analytes	Control Limits[1]
Field Parameters	Accuracy/Precision Standard Checks	pH	±0.05 pH units
		Specific Conductance	±10% RPD
		Turbidity	±2% NTU
		Dissolved Oxygen	±0.3 mg/L
		Redox Potential	±0.1 mg/L
		Temperature	±0.2°C

Note:

1. Control limits are subjected to change based on the statistical updates performed by the analytical laboratory.

TABLE 3-3
REFINED METALS SITE
SAMPLING AND ANALYSIS PROGRAM SUMMARY

Sample Location	Matrix	Parameters	Number of Samples	Field Duplicate	Matrix Spike	Blank[1]	Total Number of Samples[2]
Monitoring Wells	Groundwater (Round 1) *Two sampling rounds are proposed. If there are no volatile compounds detected during the first sampling event, this fraction will be omitted for the second event.*	Turbidity	15[3]	-	-	-	15
		pH	15[3]	-	-	-	15
		Redox potential	15[3]	-	-	-	15
		Specific Conductance	15[3]	-	-	-	15
		Temperature	15[3]	-	-	-	15
		Dissolved Oxygen	15[3]	-	-	-	15
		Volatile Organics[4]	5	1	2	2	10
		RCRA Metals plus Antimony	5	1	2	2	10
Site Soils	Soil	RCRA Metals[5]	122	12	6	14	168
NE of Production Area Soil Piles	Soil	RCRA Metals[5]	4	1	2	1	8
Off-site Soils	Soil	RCRA Metals[5]	18	1	2	2	23
Diesel Spill Soils	Soil	Organics[6]	6	1	2	2	11

TABLE 3-3
REFINED METALS SITE
SAMPLING AND ANALYSIS PROGRAM SUMMARY
(Continued)

Sample Location	Matrix	Parameter	Number of Samples	Field Duplicate	Matrix Spike	Blank[1]	Total Number of Samples[2]
Internal Building	Dust	Cadmium and Lead	3	1	2	1	7
Internal Subsurface Building	Soil	RCRA Metals[5]	45	5	6	5	61
Drainage Channel & Lined Lagoon	Sediment	RCRAMetals[5]	24	3	4	5	36

Notes
1 Blank total includes estimated number of field and equipment blanks.
2 Total number samples per event.
3 This number reflects the fewest amount of samples to be taken.
4 Volatile Organics include tetrachloroethene, 1,1,1-trichloroethane, benzene, ethylbenzene, and toluene.
5 RCRA Metals include arsenic, barium, cadmium, chromium, lead, selenium, and silver.
6 Organics include benzene, cumen, ethylbenzene, toluene, fluorene, napthalene, and phenanthrene.

TABLE 3-4
REFINED METALS SITE PROJECT ANALYTE LIST
QUANTITATION LIMITS (QL)

Metal Parameters	Method [a]	Method Detection Limit	Limit of Quantitation[b]	Laboratory Standard Operating Procedure
Aqueous				Procedure No. GR-01-121 (Preparation)
Antimony	3010A/6020	0.726 µg/l	10 µg/l	Procedure No. GR-01-129 (Analysis)
Arsenic	3010A/6020	0.338 µg/l	1.0 µg/l	
Barium	3010A/6020	0.055 µg/l	10 µg/l	
Cadmium	3010A/6020	0.01 µg/L	0.2 µg/l	
Chromium	3010A/6020	0.257 µg/l	1.0 µg/l	
Lead	3010A/6020	0.096 µg/L	1.0 µg/l	
Selenium	3010A/6020	0.621 µg/l	2.0 µg/l	
Silver	3010A/6020	0.151 µg/l	0.2 µg/l	
Mercury	7470A	0.125 µg/l	0.2 µg/l	Procedure No. GR-01-123 (Analysis)
Tetrachloroethene	5030B/8260B	0.62µg/l	1.0 µg/l	Procedure No. GR-04-104 (Preparation)
1,1,1-Trichloroethane	5030B/8260B	0.47 µg/l	1.0 µg/l	Procedure No. GR-04-104 (Analysis)
Benzene	5030B/8260B	0.77 µg/l	1.0 µg/l	
Toluene	5030B/8260B	0.61 µg/l	1.0 µg/l	
Ethylbenze	5030B/8260B	0.48 µg/l	1.0 µg/l	
Soil/Sediment				Procedure No. GR-01-103 (Preparation)
Cadmium	3050B/6020	0.001mg/kg	0.5 mg/kg	Procedure No. GR-01-0129 (Analysis)
Lead	3050B/6020	0.198 mg/kg	0.6 mg/kg	
Arsenic	3050B/6020	0.338 mg/kg	1.0 mg/kg	
Barium	3050B/6020	0.001 mg/kg	1.0 mg/kg	
Chromium	3050B/6020	0.016 mg/kg	1.0 mg/kg	
Lead	3050B/6020	0.198 mg/kg	1mg/kg	
Selenium	3050B/6020	0.01mg/kg	0.5 mg/kg	
Silver	3050B/6020	0.004 mg/kg	0.2 mg/kg	
Mercury	7471A	0.01 mg/kg	0.1 mg/kg	Procedure No. GR-04-109 (Analysis)
Benzene	5035/8260B	0.016 mg/kg	0.05 mg/kg	Procedure No. GR-04-105 (Preparation)
Toluene	5035/8260B	0.019 mg/kg	0.05 mg/kg	Procedure No. GR-04-104 (Analysis)
Ethylbenzene	5035/8260B	0.015 mg/kg	0.05 mg/kg	
Cumene	5035/8260B	0.017 mg/kg	0.05 mg/kg	
Napthalene	3550B/8270C	0.047 mg/kg	0.33 mg/kg	Procedure No. GR-09-103 (Preparation)
Fluorene	3550B/8270C	0.044 mg/kg	0.33 mg/kg	Procedure No. GR-04-103 (Analysis)
Phenanthrene	3550B/8270C	0.086 mg/kg	0.33 mg/kg	

TABLE 3-4
REFINED METALS SITE PROJECT ANALYTE LIST
QUANTITATION LIMITS (QL) (continued)

Metal Parameters	Method [a]	Method Detection Limit	Limit of Quantitation[b]	Laboratory Standard Operating Procedure
Dust				Procedure No. GR-01-103 (Preparation)
Cadmium	3050B/6020	0.001 mg/kg	0.2 mg/kg	Procedure No. GR-01-0129 (Analysis)
Lead	3050/6020	0.198 mg/kg	1 mg/kg	

NOTE:

[a] According to USEPA "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods", November 1986, SW-846, Third Edition, Update III.

[b] Specific quantitation limits are highly matrix-dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

4.0 SAMPLING PROCEDURES

Groundwater, soil, dust and sediment sampling is required to monitor the presence/absence and degree of metal constituents in these media at the Site. Specific sampling procedures are set forth in this section to meet the QA objectives outlined in Section 3.0 of this QAPP. The Site sampling Scope of Work (Section 5.0 of the RFI Work Plan) must be used concurrently with this QAPP during field sampling. SOPs are provided for the following RFI activities:

- Groundwater sampling;
- Soil sampling;
- Interior sampling (dust and soil);
- Sediment sampling;
- Field equipment decontamination; and
- Sample handling.

4.1 GROUNDWATER SAMPLING

Two groundwater sampling events will be conducted at five on-site monitoring wells.

4.1.1 Sampling Procedures

Detailed sampling procedures are provided in Section 5.2.3 of the RFI Work Plan and SOPs in Attachment B and include:

- Low-flow pump purging and sampling; and
- Field parameter measurements.

Samples will be collected directly from the low-flow pump discharge line into laboratory provided sample containers or dedicated disposable filter units and then into laboratory prepared bottles (for dissolved metal analyses). If sufficient volume is not available to sample any well with the low-flow pump, a sample will be collected using a disposable Teflon® bailer. Field parameter analyses will

include, temperature, pH, specific conductance, redox potential, dissolved oxygen and turbidity, which will be measured using flow-through cells during well purging to determine if the well was adequately purged prior to sample collection. Field blanks, equipment blanks, field duplicates, and matrix spike/matrix spike duplicate samples will be obtained as described in Section 8.1.

4.1.2 Sample Designation/Identification

Each sample will be assigned a sample designation according to a pre-determined numbering system. The sample designation at a minimum will include in abbreviated form: type of sample (i.e., MW) and a sample number. The sample designation will be written in indelible ink on an identification tag and attached to the sample container. Sample tags will also contain the items noted in Section 5.1.2.

4.1.3 Analytical Parameters

All samples collected will be analyzed for the parameters listed on Table 4-1. Table 4-1 lists the associated analytical methods, sample preservatives, sample container requirements, and holding times.

4.2 SOIL SAMPLING

Soil samples will be collected from the 57 locations. The soil sample locations are identified in Section 5.3 of the RFI Work Plan.

4.2.1 Sampling Procedures

Surface grab soil samples will be collected using a dedicated disposable plastic trowel while subsurface grab soil samples will be collected using a hand auger. The soil sample will be removed

from the hand auger using a dedicated disposable plastic scoop. The samples will be homogenized in a decontaminated stainless steel bowl and then placed directly into the laboratory supplied sample containers.

Samples collected for volatiles analysis will be obtained using Encore® samplers. Two sample aliquots will be collected from each location and placed into individual bags for shipment. A third sample aliquot will be submitted to the laboratory for moisture determination. Samples for volatiles analyses will be shipped to the laboratory within two days.

Sampling procedures are provided in detail in Section 5.3 of the RFI Work Plan and SOPs provided in Attachment B.

4.2.2 Sample Designation/Identification

Each sample will be assigned a sample designation according to a pre-determined numbering system. The sample designation at a minimum will include in abbreviated form: type of sample (i.e., S) and a sample number. The sample designation will be written in indelible ink on an identification tag and attached to the sample container. Sample tags will also contain the items noted in Section 5.1.2.

4.2.3 Analytical Parameters

All samples collected will be analyzed for the parameters listed on Table 4-1, which include cadmium and lead. Table 4-1 lists the associated analytical methods, sample preservatives, sample container requirements, and holding times.

4.3 INTERIOR SAMPLING

Floor dust samples will be collected from three locations on the floor within the Materials Storage Building and soil samples will be collected from beneath the floor slab inside the Materials Storage Building. The dust and soil sample locations are identified in Section 5.4 of the RFI Work Plan.

4.3.1 Sampling Procedures

The dust samples will be collected using dedicated disposable scoops. The soil samples will be collected using a hand auger. The soil sample will be removed from the hand auger using a dedicated disposable plastic scoop. The samples will be homogenized in a decontaminated stainless steel bowls and then placed directly into the laboratory supplied sample containers. Detailed sampling procedures are provided in Section 5.4 of the RFI Work Plan and SOPs provided in Attachment B.

4.3.2 Sample Designation/Identification

Each sample will be assigned a sample designation according to a pre-determined numbering system. The sample designation at a minimum will include in abbreviated form: type of sample (i.e., D, S) and a sample number. The sample designation will be written in indelible ink on an identification tag and attached to the sample container. Sample tags will also contain the items noted in Section 5.1.2.

4.3.3 Analytical Parameters

All samples collected will be analyzed for the parameters listed on Table 4-1, which include cadmium and lead. Table 4-1 lists the associated analytical methods, sample preservatives, sample container requirements, and holding times.

4.4 SEDIMENT SAMPLING

Composite sediment samples will be collected from two locations within the concrete and geomembrane lined surface impoundment. Sediment samples will also be collected from two former drainage channels. Figure 5-3 of the RFI Work Plan shows the proposed sediment sampling locations.

4.4.1 Sampling Procedures

Composite sediment samples will be collected over the vertical profile of sediment at each sample location within the concrete and geomembrane lined surface impoundment. Sample depths will be measured and samples will be collected using a decontaminated shovel or disposable scoop so not to jeopardize the integrity of the liner. The sediment samples collected from the drainage channels will be collected at two depth intervals (0-6" and 6-12") using a decontaminated hand auger. Each sediment sample collected will be homogenized, and placed into laboratory supplied containers and sealed. Detailed sampling procedures are provided in Section 5.5 of the RFI Work Plan and SOPs found in Attachment B.

4.4.2 Sample Designation/Identification

Each sample will be assigned a sample designation according to a pre-determined numbering system. The sample designation at a minimum will include in abbreviated form: type of sample (i.e., SED) and a sample number. The sample designation will be written in indelible ink on an identification tag and attached to the sample container. Sample tags will also contain the items noted in Section 5.1.2.

4.4.3 Analytical Parameters

Samples collected will be analyzed for the parameters listed on Table 4-1, which include cadmium and lead. Table 4-1 lists the associated analytical methods, sample preservatives, sample container requirements, and holding times.

4.5 FIELD EQUIPMENT DECONTAMINATION

To prevent possible contamination from sampling equipment, all non-dedicated sampling devices will be decontaminated. Non-dedicated equipment are the low flow pump, stainless steel bailers, mixing bowls, hand augers and shovels. Sampling equipment will be constructed of inert material (e.g., stainless steel, Teflon®). For non-dedicated equipment, field decontamination will be performed prior to its initial use, between sampling locations and between actual samples when more than one sample is to be collected at a given location. Decontamination is not required when dedicated, disposable bailers, trowels or scoops are used. All decontamination and subsequent use of decontaminated equipment will be documented in a field logbook.

All non-dedicated sampling equipment will be decontaminated according to the following procedure:

1. Wash equipment thoroughly with a low phosphate detergent (Alconox) and water using a brush to remove any particulate matter or surface film.
2. Rinse equipment with distilled water.
3. Rinse with diluted nitric acid.
4. Triple rinse with distilled water.
5. Air dry equipment.
6. Wrap equipment in a clean plastic sleeve or in aluminum foil if not used immediately.

Spent nitric acid will be contained in a bucket and placed in drums.

4.6 SAMPLE HANDLING

4.6.1 Sample Containers

Sample containers will be provided to the sampling team by the laboratory sample custodian. All sample containers used in the course of this investigation will be new containers, pre-cleaned and certified as Level II or higher by I- CHEM Inc. Certificates of analysis are available from I-CHEM upon request. All bottles will be prepared by the laboratory with the appropriate preservative. After sample collection, containers will be labeled as specified in Section 5.1.2.

4.6.2 Sample Preservation and Holding Times

The laboratory will provide appropriately prepared sample containers for this project. The sample containers will be I-Chem bottles or the equivalent which are cleaned and preserved for the specific analysis. Aqueous samples for metals analyses will be preserved with nitric acid to pH<2. Samples for dissolved metals will be field filtered prior to preservation. No acid preservation is required for the collection and analysis of soil, dust, or sediment samples. All samples will be placed on ice and maintained at a temperature of approximately 4 degrees Celsius from the time of collection to the time of analysis.

TABLE 4-1
REFINED METALS SITE
PARAMETER TABLE

Parameter	Matrix	Method	Container Type	Preservative	Holding Time
Antimony, Arsenic, Cadmium and Lead[1]	Aqueous	EPA SW-846[2] 3005A/3010A/ 6020	1 liter plastic	HNO_3 to pH<2 Cool 4°C	180 days
pH, Temperature, Redox Potential, Dissolved Oxygen, Specific Conductance	Aqueous	Manufacturer's Instructions	NA	NA	Analyze immediately
Cadmium and Lead	Soil/Sediment	EPA SW-846[2] 3050B/6010B	125 ml. amber glass	Cool 4°C	180 days
Cadmium and Lead	Dust	EPA SW-846[2] 3050B/6010B	125 ml. amber glass	Cool 4°C	180 days

Notes:

1 Includes total and dissolved metals. Dissolved metals will be filtered prior to preservation.

2 USEPA "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods", November 1986, SW-846, Third Edition Update III.

NA - Not Applicable.

5.0 CUSTODY PROCEDURES

A sample is physical evidence collected from the project site. Due to the evidential nature of the data generated from sampling, sample custody must be traceable from the time the empty sample containers are prepared by the container supplier through the reporting of the results of the analyses. As an essential part of project management, sample control procedures have been established to ensure sample integrity. All sample containers and samples will be maintained under strict custody procedures throughout the investigation. Sample custody is addressed in three parts: field sample collection, laboratory analysis and final evidence files.

A sample, sample container, or evidence file will be considered under custody if:

- the item is in actual possession of a person; or
- the item is in the view of the person, after being in actual possession of the person; or
- the item was in the person's actual physical possession but is now locked up or sealed in a tamper-proof manner; or
- the item is placed in a designated secured restricted area.

5.1 FIELD CUSTODY PROCEDURES

Sample custody for samples collected during this investigation will be maintained by the PI or field personnel collecting the samples. The PI or field personnel is responsible for documenting each sample transfer and maintaining custody of all samples until they are shipped to the laboratory or archived.

5.1.1 Field Data Documentation/Field Logs

A system of logging all pertinent data collected during sampling operations will be maintained using dedicated bound field logbooks. Each page will be numbered, dated and initialed by the person making the entry. All entries will be made in indelible ink. Incorrect entries will be crossed out with a single line and verified with the recorder's initials. At the completion of the day, if a page is not complete, a diagonal line will be drawn through the remainder of the page with the notetaker's signature at the bottom.

All sample locations will be recorded and referenced to the site map so that each location is permanently established. Samples will be tagged with all pertinent site information at the time of sampling. Section 5.1.2 describes sample identification. Pertinent site information to be supplied in the field logbook for each task is listed below:

- Signature of notetaker;
- Name and location of investigation;
- Date and time of arrival and departure;
- Names of all personnel on-site and their affiliation;
- Purpose of the visit/description of field activity;
- All field instruments used, date and time of calibration and calibration checks, method of calibration, standards used;
- All field measurement results;
- Date, time, and location of all sampling points;
- Method of sample collection;

- Any factors which could affect sample integrity;
- Name of sampler;
- Sample identification, sample description, sample preservation;
- Documentation of all conversations with the client, agency personnel, field decisions and approval; and
- Weather conditions.

Field logbooks should contain only factual information entered as real-time notes which will enable the user to recreate events on-site. They are a part of the project file and are admissible as evidence in litigation. In addition, chain-of-custody records will be prepared and kept as part of the field records.

5.1.2 Sample Identification

All sample bottles will be identified by the use of sample tags with sample identification. Each sample tag will be labeled by the sampler to avoid any possibility of sample misidentification and attached to the sample container with a wire around the container neck through a reinforced hole in the tag. Indelible ink shall be used to complete sample tags. Each sample tag will be labeled at the time of collection with, at a minimum, the following information:

- Site specific project number and name;
- Date and time (military) of sample collection;
- Sample designation (location), note here if the sample is a QC sample or to be used for QC analysis;
- Whether sample is a grab or composite;

- Presence of a preservative;
- Field representative(s) collecting the sample (Sampler); and
- Analyses requested.

The PI or field sampler will maintain custody of samples following the procedures outlined in the following section until samples are properly relinquished to the laboratory or to a common carrier for delivery to the laboratory. Once at the laboratory, each sample will be assigned a unique laboratory identification number that will be used for analysis assignment, sample tracking, and data reporting while the samples are at the laboratory.

5.1.3 Chain-of-Custody Procedures

The following chain-of-custody procedures will be used for this project:

- New, certified clean sample containers will be prepared and relinquished by the laboratory on a chain-of-custody record. The chain-of-custody record will be used for all samples collected to document the sample custody transfer from person to person.
- Any transfer of custody of containers or samples will be noted on the chain-of-custody record.
- Each sample collected for the project will be entered on the chain-of-custody record.
- The chain-of-custody will be completed as soon as possible after sample collection. The following information must be supplied to complete the chain-of-custody record:
  a. Site specific project name and number;
  b. Signature of samplers;

c. For each sample, sampling station number, date and time (military) of collection, grab or composite sample designation, and brief description of the type of sample and sampling location;

d. Number of sample containers per each sample location;

e. Analysis required;

f. Type of sample preservative;

g. Signatures of individuals involved in sample transfer (i.e., relinquishing and accepting samples). Individuals receiving the samples shall sign, date, and note the time that they received the sample on the record; and

h. Type of carrier service.

- The original chain-of-custody record will accompany the sample containers during transport to document their custody.

- If custody is relinquished through a common parcel carrier for delivery to the laboratory, the following protocol will be followed:

  a. The original completed chain-of-custody record will be placed inside the shipping package; and

  b. The shipping package will be sealed with tape and custody seals affixed. The seals will be placed on the package in such a manner that the package cannot be opened without breaking the seals. The seals will serve to document that the shipping container was not opened during the shipment through the common parcel carrier.

The chain-of-custody record is presented on Figure 5-1 of this QAPP.

5.1.4 Sample Shipment Procedures

At the end of the sampling day, all samples for chemical analysis will be packaged in shipping containers for shipment to the analytical laboratory using the following steps:

1. Check each sample bottle for a properly completed sample identification label.

2. Place sample bottles from each location in separate plastic bags, then seal.

3. Ship the samples in a large capacity (waterproof metal or equivalent strength plastic) cooler, or specific laboratory-prepared sample shipping container. Place packing material (asbestos-free vermiculite, perlite, or Styrofoam beads) on the bottom of the cooler to prevent sample bottle breakage.

4. Place sample bottles in the shipping container in a manner that they do not touch and will not touch during shipment. Secure with packing material as needed to fill void space.

5. Maintain all samples at approximately 4°C during shipment. Use ice or freezer packs to cool the samples.

6. Place the original chain-of-custody record in a plastic bag, seal, and tape it to the inside of the shipping container lid.

7. Retain the pink copy of the chain-of-custody for the QA Manager.

8. Tape cooler drain shut. Tape the cooler or shipping container closed at a minimum of two locations.

9. Place two signed and dated custody seals across each edge of the shipping container.

10. Attach completed shipping label to the top of the cooler.

11. Relinquish the cooler to the courier with the required signed and dated handbill.

12. Retain receipt of the handbill as part of the permanent documentation.

If the sample coolers are not shipped but instead picked up by the laboratory courier, step number 6 and 12 will be omitted and the chain-of-custody will be handed to and signed by the laboratory courier. The pink copy of the chain-of-custody will be maintained by the sampler and presented to the AGC Quality Assurance Manager.

5.2 LABORATORY CUSTODY PROCEDURES

Laboratory custody procedure are outlined in Attachment B, Laboratory Chain-of-Custody Standard Operating Procedure. Once the sample arrives at the laboratory, custody of the samples will be maintained by laboratory personnel. Upon receipt of the samples, the sample custody personnel will remove the chain-of-custody from the sealed cooler and sign and record the date and time on the chain-of-custody. The samples received will be verified to match those listed on the chain-of-custody. The laboratory will document and notify the Sampling Contractor's QA Manager immediately if any inconsistencies exist in the paperwork associated with the samples. The laboratory at a minimum will document the following stages of analysis: sample receipt, sample extraction/preparation, sample analysis, data reduction, and data reporting.

Samples will be given an unique laboratory identification number which is entered into the sample receiving log and the Laboratory Information Management System (LIMS). The analyst will enter the analytical data into the LIMS upon analysis completion and validation. The samples are placed into appropriate storage (refrigerators at 4°C) within an access controlled location. All samples are maintained under proper storage conditions for thirty days past the generation of the analytical report. The LIMS tracks the sample until completion of the report and invoice mailing. The data archived from the LIMS will be transferred to magnetic tape and retained for five years from the completion of sample analysis.

A chain-of-custody Sample Control Record is used as the documentation for the movement of chain-of-custody samples in and out of the access controlled storage. The analyst signs sample in and out each time a sample(s) is removed for any analysis. After all analyses are complete, the sample custodian files the form in the chain-of-custody project file. An example of the chain-of-custody Sample Control Record can be found on Figure 5-2.

Procedures for the custody of analytical data are outlined in Section 4.1, Attachment A. Sample disposal procedures are outlined in Section 4.2.4, Attachment A.

5.3 FINAL EVIDENCE FILES

The final evidence file will be a central repository for all documents which constitute evidence relevant to sampling and analysis activities as described in this QAPP. AGC is the custodian of the evidence file and maintains the contents of evident files for the investigation at the AGC Chadds Ford office. The files will be maintained as mandated by the EPA and will be maintained for a minimum six years after the termination of the order. Prior to disposal, the EPA will be offered the evidence file contents. The final evidence file will contain at a minimum the following:

- Field logbooks;
- Photographs;
- Drawings;
- Soil boring logs;
- Laboratory data deliverables;
- Data validation reports;
- Progress reports; and
- Custody documentation.

6.0 CALIBRATION PROCEDURES AND FREQUENCY

In order to provide high quality data, it is essential for all field and laboratory equipment to be in satisfactory operating condition. Thus, routine equipment calibration and maintenance is required.

6.1 FIELD INSTRUMENT CALIBRATION

During groundwater sampling, field measurements including pH, temperature, redox potential, dissolved oxygen, specific conductance and turbidity will be taken. Field calibration procedures, at a minimum, will include the following:

- Calibration of field instruments will be performed by trained technicians prior to mobilization of equipment to the site. All instruments will be calibrated as specified by the manufacturer. Standard solutions will also be checked to determine stability and operating conditions. All results of field calibrations and measurements will be maintained in bound site-dedicated logbooks assigned to the specific instrument and/or field logbooks at least daily when the instrument is in use. The recorded calibration information will include date and time of calibration, standards used, corrective actions taken if necessary, and calibration results. Routine field equipment maintenance will be documented in bound logbooks which will be kept with the field instruments.

- pH meters will be calibrated according to manufacturer's instructions prior to each use and will, at a minimum, consist of two standard buffer solutions (4, 7, or 10) obtained from chemical supply houses. Additionally, two standard buffer solutions will be analyzed as verification checks after every 20 samples and after each use. The verification check results must agree within ± 0.05 pH standard units or recalibration and reanalysis of all samples since the last verification check sample is required.

- All field thermometers will be checked against a NIST or equivalent thermometer once a year. The temperature difference will be documented in a logbook and the field measurements will be adjusted accordingly. Temperature measurements will be recorded to ± 0.2° C.

- Dissolved oxygen meters are calibrated by a trained technician prior to use in the field using a 100 percent relative humidity chamber (air calibration method). A Winkler titration is performed to check the accuracy of the air calibration method. Dissolved oxygen meters will be calibrated in the field daily by the sampling personnel using the air calibration method.

- Specific conductance meters will be calibrated prior to each use using two potassium chloride solutions prepared by a qualified laboratory or chemical supplier. These solutions will bracket the levels of the samples. At a minimum, one of the solutions will be analyzed as a verification check after each sample location and at the end of the day. The verification check must be within ± 10% of the true value. If the verification check is not within 10% of the true value, recalibration of the instrument is required and the last sample must be reanalyzed.

- Turbidity meters will be calibrated daily prior to use by using a standard of known turbidity provided by the manufacturer.

- An OVA field screening instrument will be used to monitor the head space of each monitoring well prior to sampling. The OVA will be calibrated by a trained field technician with a certified gas (86.5 ppm methane in air) prior to mobilization of equipment to the Site. Daily calibrations on-site will be performed by the field sampling personnel. Initial calibration information will be recorded in field logbooks.

All calibration procedures performed will be documented in the field logbook and will include the date and time of calibration, name of the person performing the calibration, reference standards used, and the instrument readings.

6.2 LABORATORY INSTRUMENT CALIBRATION

Calibration procedures for a specific laboratory instrument will consist of initial calibrations, initial calibration verifications and continuing calibration verification. The SOP for each analysis performed in the laboratory describes the calibration procedures, their frequency, acceptance criteria and the conditions that will require recalibration. In all cases, the initial calibration will be verified using an independently prepared calibration verification solution. Specific laboratory instrument calibration requirements summarized in Table 6-1 outlined in Section 13.0 of each applicable laboratory SOP provided in Attachment B.

The laboratory maintains a sample logbook for each instrument. These logbooks contain the following information: instrument identification, date of calibration, analyst, calibration standards, and samples associated with these calibrations.

If equipment fails calibration or equipment malfunction is noted during calibration, the equipment is tagged and removed from service. The equipment is held out of service until repairs and successful calibration occur. All malfunctions, repairs and recalibrations are recorded in the appropriate instrument maintenance and calibration logs.

TABLE 6-1
SUMMARY OF LABORATORY CALIBRATION REQUIREMENTS

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria
SW8260B	Volatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥0.30[c] and %RSD for RFs for CCCs ≤30% and one option below (No RF <0.05)
				Option 1 Linear-mean RSD for all analytes ≤15% with no individual analyte RSD >30%
				Option 2 Linear-least squares regression r>0.995
				Option 3 Non-linear-COD ≥0.990 (6 points shall be used for second order, 7 points shall be used for third order)
		Second-source calibration verification	Once per five-point initial calibration	All analytes within ±25% of expected value
SW8260B	Volatile Organics	Continuing calibration check	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥0.30[c]; and CCCs ≤20% difference (when using RFs) or drift (when using least squares regression or non-linear calibration)

TABLE 6-1
SUMMARY OF LABORATORY CALIBRATION REQUIREMENTS
(Continued)

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria
				All calibration analytes within ±20% of expected value

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria
SW8270C	Semivolatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥0.30[c] and %RSD for RFs for CCCs ≤30% and one option below (No RF <0.05)
				Option 1 Linear-mean RSD for all analytes ≤15% with no individual analyte RSD >30%
				Option 2 Linear-least squares regression r>0.995
				Option 3 Non-linear-COD ≥0.990 (6 points shall be used for second order, 7 points shall be used for third order)
		Second-source calibration verification	Once per five-point initial calibration	All analytes within ±25% of expected value

TABLE 6-1
SUMMARY OF LABORATORY CALIBRATION REQUIREMENTS
(Continued)

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria
		Calibration verification	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥0.050; and CCCs ≤20% difference (when using RFs) or drift (when using least squares regression or non-linear calibration)
				All calibration analytes within ±20% of expected value

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria
SW6020	ICP/MS Metals	MS tuning sample	Prior to initial calibration and calibration verification	SW6020 paragraph 5.8
		Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis	N/A
		Calibration blank	Before beginning a sample run, after every 10 samples and at end of the analysis sequence	No analytes detected ≥RL
		Calibration verification (second source standard)	Before beginning a sample run, after every 10 samples and at the end of the analysis sequence	All analyte(s) within ±10% of expected value

7.0 ANALYTICAL PROCEDURES

7.1 FIELD ANALYTICAL PROCEDURES

Temperature, redox potential, dissolved oxygen, turbidity, pH and specific conductance measurements of samples will be performed to determine if a well has been adequately purged as described in Section 5.2 of the RFI Work Plan. All field measurements will be collected according to manufacturer's instructions and the SOPs provided in Attachment B. Table 3-2 presents the quality control requirements and criteria for the field measurement parameters.

7.2 LABORATORY ANALYTICAL PROCEDURES

All sample media will be analyzed by TriMatrix Laboratories, Inc. TriMatrix is located at:

5555 Glenwood Hills Parkway SE
Grand Rapids, Michigan 49588
Telecon (616) 975-4500
Facsimile (616) 942-7463

The laboratory will conduct the analyses in accordance with the specified methods in Table 7-1 Soil and sediment samples will be dried prior to digestion. Only the most updated U.S. EPA approved SW-846 methodology will be used. These methods have been selected because they are deemed sufficient to achieve the project data quality objectives. Standard Operating Procedures for the analyses are identified in Table 7-1 and are provided as attachments to this document. These SOPs for sample preparation and analysis are based on SW-846, third edition, updated through June 1997. These SOPs provide sufficient detail and are specific to this investigation.

The laboratory SOPs listed in Table 7-1 include a QA section which address the minimum QC requirements for analyses. All quality control samples identified in Section 8.0 will be analyzed as appropriate for each method. The quality control criteria as identified in the referenced U.S. EPA Methods must be met or appropriate action will be taken. This may include termination of analysis, reanalysis of samples, or accepting the data and acknowledging a level of uncertainty.

7.2.1 Project Target Compounds and Laboratory Detection Limits

A complete listing of the project target analytes, quantitation limits and laboratory method detection limits is provided in Table 3-4.

7.2.2 List of Associated Quality Control Samples

Section 13.0 of the laboratory SOPs listed in Table 7-1 specifies the minimum QC requirements for the analysis of specific analyte groups. All project target analytes will be added to the spiking solution, in compliance with project requirements. Section 8.0 of this QAPP contains a complete listing of the associated QC samples for every analyte group and matrix.

TABLE 7-1
REFINED METALS SITE
PROJECT ANALYTE LIST

Metal Parameters	Method [b]	Laboratory Standard Operating Procedure
Aqueous[a]		
RCRA Metals except Mercury	3010A	Procedure No. GR-01-121 (Preparation)
RCRA Metals except Mercury	6020	Procedure No. GR-01-129 (Analysis)
Mercury	7470	Procedure No. GR-01-123 (Analysis)
Volatiles	5030B	Procedure No. GR-04-104 (Preparation)
Volatiles	8260B	Procedure No. GR-04-104 (Analysis)
Soil/Sediment/Dust		
RCRA Metals except Mercury	3050B	Procedure No. GR-01-103 (Preparation)
RCRA Metals except Mercury	6020	Procedure No. GR-01-100 (Analysis)
Mercury	7470	Procedure No. GR-01-109 (Analysis)
Volatiles	8260B	Procedure No.GR-04-104 (Preparation)
Semivolatiles	3550B	Procedure No.GR-09-103 (Preparation)
Semivolatiles	8270C	Procedure No. GR-04-103 (Analysis)

NOTE:

a. Includes dissolved and total metals.

b. According to USEPA "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods", November 1986, SW-846, Third Edition, Update III.

8.0 INTERNAL QUALITY CONTROL CHECKS

Quality control and quality assurance procedures include both field and laboratory check samples and are designed to ensure and document the overall quality of the data. QA/QC checks detect potential problems at the source and, if necessary, trace the sample analytical pathways for introduction of contamination. The quality control data generated in the field will monitor sampling techniques, reproducibility, and cleanliness. Quality control data generated by the laboratory will monitor reproducibility (precision), cleanliness, and accuracy in analyzed samples. During data validation, QC check results are used to evaluate precision, accuracy, and representativeness of the overall sampling and analytical program.

8.1 FIELD QUALITY CONTROL CHECKS

The field quality control samples monitor the data quality as it is affected by the field procedures and conditions. Field QC samples are control samples that are introduced to the laboratory from the field. During field sampling efforts, different types of QA samples will be collected: field blanks, equipment blanks, field replicate and samples, matrix spike/matrix spike duplicate samples. The QC criteria for each field quality control sample are provided in Table 3-2. Validation guidelines outlined in Section 9.2 will be used for the acceptance limits of the field QC samples. Each type of QA sample is described below.

8.1.1 Field Blanks

Field blanks are collected in the field by pouring demonstrated analyte-free water provided from the laboratory from one sample container into a preserved sample container identical to those provided for sample collection. One field blank will be collected for each sampling round, and will be

analyzed for the same parameters as the actual samples. Field blanks for dissolved metals will be filtered through a 0.45 μm filter prior to preservation.

8.1.2 Equipment Blanks

Equipment blanks are prepared in the field to ensure a sampling device (e.g., pump, bailer, bailer line) has been effectively cleaned. The sampling equipment is filled with deionized water or deionized water is pumped through the device, transferred to the laboratory supplied sample bottles, preserved if necessary, and sent to the laboratory for analyses with the site samples. If dedicated equipment is not used, one equipment blank will be submitted for analyses for every 10 samples per media collected, and will be analyzed for the same parameters as the field samples. Equipment blanks for dissolved metals will be filtered through a 0.45 μm filter prior to preservation.

8.1.3 Field Duplicate Samples

Field duplicate samples consist of an actual sample taken in the field which has been split into two aliquots and put into two separate sampling containers. Aqueous samples will be obtained by alternately filling sample containers from the same sampling device for each parameter. When obtaining soil, dust, or sediment duplicate samples, homogenization of the sample aliquot prior to filling the sample containers will be performed to generate two equally representative samples. The samples will be transported to the laboratory and analyzed as two separate samples. The results will be used to assess laboratory accuracy and precision of sampling and analysis.

Each sample will be labeled with a unique sample number and submitted to the laboratory for the appropriate analyses. Field duplicate samples determine analytical precision and sample representativeness. One field duplicate sample will be collected for every 10 samples per media collected.

8.1.4 Matrix Spike/Matrix Spike Duplicate Samples

Matrix spike (MS) and matrix spike duplicate (MSD) samples will be submitted in association with metal analyses as further QC checks. MS and MSDs will be collected from the same location as the field sample and in the same manner.

Each sample will be labeled with the sample number as the original sample, designated as MS or MSD sample, and submitted to the laboratory for the appropriate analyses. MS/MSD samples determine accuracy by the recovery rates of the compounds added by the laboratory (all site related metal compounds will be included in the spiking solution). The MS/MSD samples also monitor any possible matrix effects specific to samples collected from the site and the extraction/digestion efficiency. In addition, the analysis of MS and MSD samples check precision by comparison of the two spike recoveries. One MS and MSD sample will be collected for every 20 samples per media collected and analyzed.

8.2 LABORATORY QUALITY CONTROL CHECKS

All QC procedures employed by the laboratory will be, at a minimum, equivalent to those required in the specified analytical methods. Laboratory QC checks are accomplished through the analyses of laboratory blanks, matrix spike/matrix spike duplicates, calibration verifications, laboratory fortified blanks and performance evaluation samples. When internal quality control results fall

outside method acceptance criteria, the data will be reported, and the analysis repeated, flagged or accepted according to the specified analytical methods. The following sections generally describe internal laboratory quality control check samples. Quality control requirements are outlined in Section 18 of the laboratory SOPs.

8.2.1 Laboratory Blanks

Method/preparation blanks are generated within the laboratory during the processing of the actual samples. These blanks will be prepared using the same reagents and procedures and at the same time as the project samples are being analyzed. If contamination is found in the method blank, it indicates that similar contamination found in associated samples may have been introduced in the laboratory and not actually present in the samples themselves. Guidelines for accepting or rejecting data based on the level of contamination found in the blank are presented in the specified analytical method and laboratory SOPs.

A minimum of one method blank per 20 samples will be analyzed or, in the event that an analytical round consists of less than 20 samples, one method blank sample will be analyzed.

8.2.2 Instrument Blanks

Instrument blanks are prepared by the laboratory using deionized water for sample analysis. Instrument blanks are analyzed every ten samples to verify no cross contamination or baseline drifting has occurred. An instrument blank is generally analyzed after each calibration verification standard.

8.2.3 Matrix Spike/Matrix Spike Duplicates

Matrix spike analyses are performed in association with the sample metal analyses. Matrix spikes are prepared by placing a known quantity of selected target analytes into a second aliquot of an actual field sample (Section 9.1.4). All project target analytes will be included in the spiking solution. The spiking occurs prior to sample preparation and analysis. The matrix spike is then processed in a manner identical to the field sample. Recovery of each of the spiked compounds reflects the ability of the laboratory and method to accurately determine the quantity of that compound in that particular sample.

Matrix spike duplicates are identical to matrix spikes. Another aliquot of the field sample used for the MS is fortified with the same quantity of the spiking compounds and is processed in an identical manner. In addition to providing a measure of accuracy of the determination, the results for the MS/MSD pair provide a measure of precision of the determinations by assuring the availability of positive results for comparison.

Matrix spike and matrix spike duplicates will be analyzed at a frequency of one pair per sample delivery group of up to 20 samples per media collected.

8.2.4 Calibration Verifications

Initial calibration of the instruments will be completed prior to sample analysis following the specified analytical methods and laboratory SOPs. Additionally, continuing calibration standards will be analyzed at least every tenth sample. Recalibration is required if the continuing calibration standards do not meet U.S. EPA method criteria. Specific calibration standard procedures are outlined in the laboratory Standard Operating Procedures (Attachment B).

8.2.5 Laboratory Control Sample (LCS)

The LCS is prepared by the laboratory by adding analytes of known concentrations to DI water for aqueous metals analysis. Reference materials with known concentrations are digested concurrent with samples for solid metals analyses. The LCS is designed to assess the capability of the laboratory to perform the analytical methods. If the analytes present in the LCS are not recovered within the criteria defined in the specified analytical methods, the samples will be redigested and reanalyzed or data will be flagged.

8.2.6 Performance Evaluation Samples

Performance evaluation (PE) samples are of known composition which has been provided to the laboratory for analysis by either an agency or client. The laboratory results are compared to the actual values to evaluate the laboratory's performance. Performance evaluation sample analyses are performed on a regular basis as required for the laboratory's certifications. Some PE programs which TriMatrix participates in are USEPA Water Pollution Performance Evaluation Study, ASI National Performance Evaluation Study and USEPA Water Supply Study.

9.0 DATA REDUCTION, VALIDATION AND REPORTING

9.1 DATA REDUCTION

9.1.1 Field Data Reduction Procedures

All field data will be written in ink into bound field logbooks immediately after measurements are taken. If errors are made, the error will be crossed out with a single line, initialed and dated with the corrections written clearly adjacent to the original entry.

9.1.2 Laboratory Data Reduction Procedures

All analytical data will be permanent, complete and retrievable. The analyst will record the analytical data in notebooks along with other pertinent information such as the laboratory ID number. Each page of the notebook shall be signed and dated by the analyst. Periodic review of the notebooks will be performed by a supervisor prior to final data reporting. Upon analysis completion and laboratory validation, the analyst will enter the analytical data into the LIMS.

The laboratory will report sample results on analysis report forms and provide the information described in USEPA SW-846 for all analyses for each package. A CLP-like data deliverables package is required. All laboratory data will undergo the data validation procedures described in the Laboratory QA Manual prior to final reporting. Data will be stored on the laboratory's network until the investigation is complete. Data archived from the LIMS will be transferred to magnetic tape which will be retained by the laboratory an additional five years, minimum.

The equations that will be employed in reducing data are presented in Section 16 of the associated SOPs. The formulas included in the SOP make pertinent allowances for sample matrices. All calculations are checked by a second person prior to data entry into the LIMS. All groundwater metals results will be reported in micrograms per liter (μg/L). Soil and sediment metal results will be reported, corrected for moisture content, in mg/kg. Dust metals results will be reported on an "as received basis" in mg/kg. All blank results and QC data will be included in the data deliverables/package. Blank results will not be subtracted from the sample results. The blank results and QC data will be used in data validation to review sample results qualitatively. Data validation will be performed in general accordance with the guidelines identified in Section 9.2. Outliers and other questionable data will be addressed in the data validation report and specific QA/QC flags will be applied to questionable data. The QA/QC flags will be consistent with the USEPA data validation guidelines.

9.2 DATA VALIDATION

9.2.1 Procedures Used to Validate Field Data

Validation of the field data will be performed by the PI under the supervision of the QA manager. One hundred percent of the field analytical data will be validated. The procedures to validate the field dat for this investigation include checking for transcription errors and review of logbook, on part of the field crew members. This task will be the responsibility of the PI.

9.2.2 Procedures Used to Validate Laboratory Data

Validation of analytical data as received from the laboratory will be performed by the AGC Quality Assurance Manager or Quality Assurance Scientist. Validation will be performed on 100% of the

analytical data in general accordance with the following data validation guidance document, where applicable: USEPA Contract Laboratory Program National Functional Guidelines for Organic (and Inorganic) Data Review, Office of Emergency and Remedial Response, USEPA, Washington, D.C. February 1994. The Data Management Plan, provided as Attachment C, discusses the specific procedures for the validation of CLP data. Quality control requirements specified in the methods will also be used to evaluate the data. Specific data validation procedures are outlined in Tables 9-1 through 9-3 validation criteria are not met for any parameter, the associated samples will be qualified as indicated in Tables 9-1 through 9-3.

The following presents definitions for the validation qualifiers:

U	-	The analyte was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
J	-	The associated value is an estimated quantity.
R	-	The data are unusable. (Note: The analyte may or may not be present.)
UJ	-	The analyte was analyzed for, but was not detected. The associated detection limit is an estimate and may be inaccurate or imprecise.

The purpose of data validation is to assess the usability of the data by determining if the laboratory analyses met the PARCC criteria set by the site DQO's, the analytical method used and the guidance documents. Upon completion of data validation, the existing results will be reported in tabular form with data validation flags applied as appropriate to determine the usefulness of the data. The data validation flags will be consistent with the USEPA data validation guidelines. A data validation report will be written to assist in making decisions based on the analytical results.

9.3 DATA REPORTING

Data validation reports, along with copies of all support documentation, validated data summary tables, and analytical data packages, will be submitted monthly to RMC Project Manager as data is validated. The RMC Project Manager will forward to the EPA, after adequate time for review, all documents, data and reports. The data validation report will be prepared using USEPA's Region V format.

9.3.1 Field Data Reporting

Field data reporting will be conducted through the transmission of logbook sheets containing tabulated results of all measurements made in the field, and documentation of all field activities.

9.3.2 Laboratory Data Reporting

The task of reporting laboratory data (to the U.S. EPA) begins after the independent validation activity has been concluded. The AGC Quality Assurance Manager must perform a final review of the report summaries and case narratives to determine whether the report meets project requirements. In addition to the record of chain-of-custody, the report format shall consist of the following:

1. Case Narrative:
    i. Date of issuance
    ii. Laboratory analysis performed
    iii. Any deviations from intended analytical strategy
    iv. Laboratory batch number

v. Numbers of samples and respective matrices

vi. QC procedures utilized and also references to the acceptance criteria

vii. Laboratory report contents

viii. Project name and number

ix. Condition of samples 'as-received'

x. Discussion of whether or not sample holding times were met

xi. Discussion of technical problems or other observations which may have created analytical difficulties

xii. Discussion of any laboratory QC checks which failed to meet project criteria

xiii. Signature of the Laboratory QA Manager

2. Chemistry Data Package:

i. Case narrative for each analyzed batch of samples

ii. Summary page indicating dates of analyses for samples and laboratory QC checks

iii. Cross referencing of laboratory sample to project sample identification numbers

iv. Description of data qualifiers to be used

v. Sample preparation and analyses for samples

vi. Sample results

vii. Raw data for sample results and laboratory QC samples

viii. Results of (dated) initial and continuing calibration checks, and GC/MS tuning results

ix. MS/MSD recoveries, laboratory control samples, method blank results, calibration check compounds, and system performance check compound results

x. Labeled (and dated) chromatograms/spectra of sample results and laboratory QC checks

xi. Copies of Nonconformance Reports.

The data package submitted will be a "CLP-like" data package consisting of all the information presented in a CLP data package (but without the CLP forms).

All deliverables/packages from each laboratory must be paginated in ascending order. The laboratory must keep a copy of the paginated package in order to be able to respond efficiently to data validation inquiries. Any errors in reporting identified during the data validation process must be corrected by the laboratory as requested. All data validation inquiries to the laboratory must be addressed by a written response from the laboratory in question.

The deliverables will be provided to the AGC Quality Assurance Manager and will be made available to the EPA upon request.

9.4 DATA ACQUISITION REQUIREMENTS AND DATA QUALITY MANAGEMENT

Once the samples are collected and sent to the laboratory, the field PI will send a copy of the chain-of-custody and field notes to the AGC Quality Assurance Manager. The chain-of-custodies will be checked for the appropriate analytical methods defined, parameters requested, number of samples collected and QC samples collected. The laboratory will be contacted if any information on the chain-of-custody is missing or incorrect. The CLP-like deliverables hard copy and electronic data will be provide to the AGC QA Manager. The QA Manager will perform an initial check to verify that all the samples were analyzed, the correct methods were used for analyses, all requested

parameters were analyzed and samples were analyzed within the holding time requirements. The electronic deliverables will be downloaded into a site specific database and checked with the hard copy deliverables during the data validation process. A project status form will be completed each time a check level is performed. The project status form and check forms are included in Attachment D.

Analytical data, reports, and any other project related information produced during this project will be retained by AGC or its designee. Project reports, tables, etc. may be stored in project specific electronic files. On a regular basis, the data will be backed up on magnetic tapes and stored off-site. The files will be maintained as mandated by the EPA and will be maintained for a minimum six years after the termination of the order. Prior to disposal, the EPA will be offered the evidence file contents.

TABLE 9-1
SUMMARY OF DATA VALIDATION REQUIREMENTS FOR METHOD SW8260B

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Qualifier Requirements
SW8260B	Volatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥0.30[c] and %RSD for RFs for CCCs ≤30% and one option below (No RF <0.05)	If %RSD not met, apply "J" to positive results for samples associated with the calibration
				Option 1 Linear-mean RSD for all analytes ≤15% with no individual analyte RSD >30%	If RF <0.05, apply "J" to positive results for samples associated with the calibration. Apply "R" to non-detected results
				Option 2 Linear-least squares regression r>0.995	
				Option 3 Non-linear-COD ≥0.990 (6 points shall be used for second order, 7 points shall be used for third order)	
		Second-source calibration verification	Once per five-point initial calibration	All analytes within ±25% of expected value	Apply "J" to positive results for specific analyte(s) for all samples associated with the calibration; "UJ" to non-detects

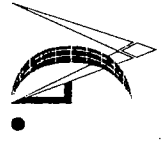


TABLE 9-1
SUMMARY OF DATA VALIDATION REQUIREMENTS FOR METHOD SW8260B
(Continued)

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Qualifier Requirements
SW8260B	Volatile Organics	Continuing calibration check	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥0.30[c]; and CCCs ≤20% difference (when using RFs) or drift (when using least squares regression or non-linear calibration)	If %RSD not met, apply "J" to positive results for samples associated with the calibration
				All calibration analytes within ±20% of expected value	If %RSD not met, apply "J" to positive results for samples associated with the calibration
		ISs	Immediately after or during data acquisition for each sample	Retention time ±30 seconds from retention time of the mid-point std. in the ICAL EICP area within -50% to +100% of ICAL mid-point std.	Apply "J" to all results for analytes associated with the IS. Apply "UJ" to non-detect results
		Blank (Laboratory method or field)	Per QAPP	No common contaminant >5X QL; no other analytes detected ≥QL	Qualify associated samples. Present QL and apply "U" for results less than QL. Present result and apply "U" for results greater than QL

TABLE 9-1
SUMMARY OF DATA VALIDATION REQUIREMENTS FOR METHOD SW8260B
(Continued)

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Qualifier Requirements
SW8260B	Volatile Organics	LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, specified in Table 3-3	For specific analyte(s) in all samples in the associated analytical batch if the LCS %R >UCL, apply "J" to all positive results if the LCS %R <LCL, apply "J" to all positive results, apply "UJ" to all non-detects
		MS/MSD	One MS/MSD per every 20 samples per matrix	QC acceptance criteria, specified in Table 3-3	None
		Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to criteria listed in the method.	Apply "R" to all results for all samples associated with the tune

TABLE 9-1
SUMMARY OF DATA VALIDATION REQUIREMENTS FOR METHOD SW8260B
(Continued)

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Qualifier Requirements
SW8260B	Volatile Organics	Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria specified in lab SOP	For the samples; if the %R >"UCL" for a surrogate, apply "J" to all positive results if the %R <LCL for a surrogate, apply "J" to all positive results; apply "UJ" to all non-detect results if any surrogate recovery is <10%, apply R to non-detect results
		Qualitative Identification	All sample analyses	Acceptance criteria specified in lab SOP	Apply "R" to results > QL; represent QL for results <QL.

Qualifier requirements are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed

TABLE 9-2
SUMMARY OF DATA VALIDATION REQUIREMENTS FOR METHOD SW8270C

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Qualifier Requirements
SW8270C	Semivolatile Organics	Five-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥0.30[c] and %RSD for RFs for CCCs ≤30% and one option below (No RF <0.05)	Apply "J" to positive results for specific analyte(s) for all samples associated with the calibration;"UJ" to non-detects
				Option 1 Linear-mean RSD for all analytes ≤15% with no individual analyte RSD >30%	If RF <0.05, apply "J" to positive results for samples associated with the calibration. Apply "R" to non-detected results
				Option 2 Linear-least squares regression r>0.995	
				Option 3 Non-linear-COD ≥0.990 (6 points shall be used for second order, 7 points shall be used for third order)	

TABLE 9-2
SUMMARY OF DATA VALIDATION REQUIREMENTS FOR METHOD SW8270C
(Continued)

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Qualifier Requirements
SW8270C	Semivolatile Organics	Second-source calibration verification	Once per five-point initial calibration	All analytes within ±25% of expected value	Apply "J" to positive results for specific analyte(s) for all samples associated with the calibration; "UJ" to non-detects
		Calibration verification	Daily, before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥0.050; and CCCs ≤20% difference (when using RFs) or drift (when using least squares regression or non-linear calibration)	If %RSD not met, apply "J" to positive results for samples associated with the calibration
				All calibration analytes within ±20% of expected value	If %RSD not met, apply "J" to positive results for samples associated with the calibration
		ISs	Immediately after or during data acquisition for each sample	Retention time ±30 seconds from retention time of the mid-point std. in the ICAL EICP area within -50% to +100% of ICAL mid-point std.	Apply "J" to all results for analytes associated with the IS "UJ" non-detect results

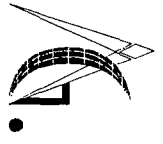


TABLE 9-2
SUMMARY OF DATA VALIDATION REQUIREMENTS FOR METHOD SW8270C
(Continued)

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Qualifier Requirements
SW8270C	Semivolatile Organics	Blank (Laboratory method or field)	One per analytical batch	No common contaminant >5X QL; no other analytes detected ≥QL	Qualify associated samples. Present QL and apply "U" for results less than QL. Present result and apply "U" for results greater than QL
		LCS for all analytes	One LCS per analytical batch	QC acceptance criteria, specified in Table 3-3	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R >UCL, apply J to all positive results if the LCS %R <LCL, apply J to all positive results, apply UJ to all non-detects
		MS/MSD	One MS/MSD per every 20 samples per matrix	QC acceptance criteria, specified in Table 3-3	None
		Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to criteria listed in the method	Apply R to all results for all samples associated with the tune

TABLE 9-2
SUMMARY OF DATA VALIDATION REQUIREMENTS FOR METHOD SW8270C
(Continued)

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Qualifier Requirements
SW8270C	Semivolatile Organics	Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria specified in lab SOP	For the samples; if the %R >"UCL" for a surrogate, apply "J" to all positive results if the %R <LCL for a surrogate, apply "J" to all positive results; apply "UJ" to all non-detect results if any surrogate recovery is <10%, apply R to non-detect results
		Qualitative identification	All sample analyses	Acceptance criteria specified in lab SOP	Apply "R" to results >QL: present QL for results <QL.

Requirements are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed

TABLE 9-3
SUMMARY OF DATA VALIDATION REQUIREMENTS FOR METHOD SW6020

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Qualifier Requirements
SW6020	ICP/MS Metals	MS tuning sample	Prior to initial calibration and calibration verification	SW6020 paragraph 5.8	Apply R to all results for all analytes for all samples associated with the MS tuning
		Initial calibration (minimum 1 standard and a blank)	Daily initial calibration prior to sample analysis	N/A	Apply R to all results for specific analyte(s) for all samples associated with the calibration if calibration not done
		Calibration blank	Before beginning a sample run, after every 10 samples and at end of the analysis sequence	No analytes detected ≥RL	Qualify associated samples with results less than 5 X blank level. Present result and apply "U".
		Calibration verification (second source standard)	Before beginning a sample run, after every 10 samples and at the end of the analysis sequence	All analyte(s) within ±10% of expected value	Apply "J" to positive results for specific analyte(s) for all samples associated with the calibration;"UJ" to non-detects

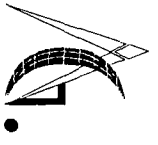


TABLE 9-3
SUMMARY OF DATA VALIDATION REQUIREMENTS FOR METHOD SW6020
(Continued)

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Qualifier Requirements
SW6020	ICP/MS Metals	Method blank	One per analytical batch	No analytes detected ≥RL	Qualify associated samples with results less than 5 X blank level. Present result and apply "U"
		Interference check solutions (ICS-A and ICS-AB)	At the beginning and end of an analytical run or twice during an 12 hour period, whichever is more frequent	ICS-A All non-spiked analytes ≤RL ICS-AB Within ±20% of true value	Apply "J" to all results for specific analyte(s) in associated samples with concentrations interfering elements >50% level for ICS
		LCS for the analyte	One LCS per analytical batch	QC acceptance criteria, Table 3-2	For specific analyte(s) in all samples in the associated analytical batch; if the LCS %R >UCL, apply "J" to all positive results if the LCS %R <LCL, apply "J" to all positive results, apply "UJ;" if %R ≥30% R if <30% to all non-detects

TABLE 9-3
SUMMARY OF DATA VALIDATION REQUIREMENTS FOR METHOD SW6020
(Continued)

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Qualifier Requirements
SW6020	ICP/Metals	Dilution test	Each preparatory batch	1:4 dilution must agree within ±10% of the original determination	Apply J to all sample results if post digestion spike test not performed
		Post digestion spike addition	When dilution test fails	Recovery within 75-125% of expected results	Apply J to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post digestion spike addition
		MS/MSD	One MS/MSD per every 20 Air Force project samples per matrix	QC acceptance criteria, Table 3-3	For the specific analyte(s) in all associated samples if; "J" positive results; "1" "UJ" nondetect if %R <LCL.

TABLE 9-3
SUMMARY OF DATA VALIDATION REQUIREMENTS FOR METHOD SW6020
(Continued)

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Qualifier Requirements
SW6020	ICP/MS Metals	Internal Standards (ISs)	Every sample	IS intensity within 30-120% of intensity of the IS in the initial calibration	Apply "J" to all results for specific analyte(s) in all samples associated with the IS. Apply "UJ" to non-detects

Qualifier requirements are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed

10.0 PERFORMANCE AND SYSTEM AUDITS AND FREQUENCY

Performance and system audits of both the laboratory and field operations will be conducted to verify that sampling and analysis are performed in accordance with the procedures established in this QAPP.

10.1 FIELD PERFORMANCE AND SYSTEM AUDITS

10.1.1 Internal Field Audits

10.1.1.1 **Internal Field Audit Responsibilities**

Internal audits of field activities including sampling and field measurements will be conducted by the AGC QA Manager. These audits will verify that all established procedures are being followed.

10.1.1.2 **Internal Field Audit Frequency**

Internal field audits will be conducted once at the beginning of the Site sample collection activities.

10.1.1.3 **Internal Field Audit Procedures**

The audits will include an examination of field sampling records, field screening analytical results, field instrument operating records, sample collection, handling and packaging in compliance with the established procedures, and maintenance of QA procedures, chain-of-custody, etc. Follow-up audits will be conducted to correct any deficiencies and to verify that QA procedures are maintained throughout the investigation. The audits will involve a review of field measurement records,

instrumentation calibration records, and sample documentation. The field audit checklist to be used for this project is provided as Attachment E of this QAPP. The field audit procedures applicable to the audit must exceed 90 percent positive (proper) responses to meet the acceptance criteria.

10.1.2 External Field Audits

10.1.2.1 **External Field Audit Responsibilities**

External field audits may be conducted by the U.S. EPA RCRA Permit Writer/Project Manager.

10.1.2.2 **External Field Audit Frequency**

External field audits may be conducted any time during the field operations. These audits may or may not be announced and are at the discretion of U.S. EPA.

10.1.2.3 **External Field Audit Process**

External field audits will be conducted according to the field activity information presented in the QAPP. The external field audit process can include (but not be limited to): sampling equipment decontamination procedures, sample bottle preparation procedures, sampling procedures, examination of field sampling and safety plans, sample vessel cleanliness and QA procedures, procedures for verification of field duplicates, sample preservation and preparation for shipment, as well as field screening practices.

10.2 LABORATORY PERFORMANCE AND SYSTEMS AUDITS

10.2.1 Internal Laboratory Audits

10.2.1.1 **Internal Laboratory Audit Responsibilities**

The internal laboratory audit will be conducted by the AGC QA Manager.

10.2.1.2 **Internal Laboratory Audit Frequency**

The internal system audits will be done on an annual basis while the internal performance audits will be conducted on a quarterly basis. The laboratory regularly participates in performance evaluation audits as part of their laboratory certification efforts. Such performance evaluation audits which TriMatrix participates in are the USEPA Water Study, USEPA Water Pollution Performance Evaluation Studies, and the ASI National Performance Evaluation Study. AGC performed its last audit of the laboratory in May 1998 to observe conformance to USEPA inorganic methods.

10.2.1.3 **Internal Laboratory Audit Procedures**

The internal system audits will include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation and analysis, instrument operating records, etc.

The performance audits will involve preparing blind QC samples and submitting them along with project samples to the laboratory for analysis throughout the project. The AGC QA Manager will evaluate the analytical results of these blind performance samples to ensure the laboratory maintains acceptable QC performance.

10.2.2 External Laboratory Audits

10.2.2.1 **External Laboratory Audit Responsibilities**

An external audit will be conducted as required by appropriate QA staff of the Waste, Pesticides and Toxics Division, U.S. EPA Region 5.

10.2.2.2 **External Laboratory Audit Frequency**

An external audit will be conducted at least once prior to the initiation of the sampling and analysis activities. These audits may or may not be announced and are at the discretion of the U.S. EPA. EPA Region 5 performed its last direct audit of TriMatrix in 1994 while TriMatrix was still a part of WW Engineering & Science.

10.2.2.3 **Overview of the External Laboratory Audit Process**

External audits may include any or all of: review of laboratory analytical procedures, laboratory on-site visits, and/or submission of performance evaluation samples to the laboratory for analysis. Failure of any or all audit procedures chosen can lead to laboratory disqualification and the requirement that another suitable laboratory be chosen.

An external on-site review can consist of: sample receipt procedures, custody and sample security and log-in procedures, sample through put tracking procedure, review of instrument calibration records, instrument logs and statistics (number and type), review of QA procedures, log books, sample prep procedures, sample analytical SOP review, instrument (normal or extended quantitation report) reviews, personnel interviews, review of deadlines and glassware prep, and a close out to offer potential corrective action.

It is common practice when conducting an external laboratory audit to review one or more data packages from sample lots recently analyzed by the laboratory. This review will most likely include but not be limited to:

- Comparison of resulting data to the SOP or method, including coding for deviations.
- Verification of initial and continuing calibrations within control limits.
- Verification of surrogate recoveries and instrument timing results where applicable.
- Review of extended quantitation reports for comparisons of library spectra to instrument spectra, where applicable.
- Recoveries on controls standard runs.
- Review of run logs with run times, ensuring proper order of runs.
- Review of spike recoveries/QC sample data.
- Review of suspected manually integrated GC data and its cause (where applicable).
- Review of GC peak resolution for isolated compounds as compared to reference spectra (where applicable).
- Assurance that samples are run within holding times.

Ideally, the data should be reviewed while on the premises, so that any data called into question can be discussed with the staff.

11.0 PREVENTATIVE MAINTENANCE

11.1 FIELD INSTRUMENT PREVENTATIVE MAINTENANCE

Field measurement equipment, OVA, pH meters, thermometers, dissolved oxygen meters, and specific conductance meters will be maintained in accordance with manufacturer's instructions. All field equipment will be checked by qualified technicians prior to use in the field. The instrument operator will be responsible for ensuring that the equipment is operating properly prior to use in the field. Any problems encountered while operating the instrument will be documented in the field logbook. Critical spare parts such as batteries and pH probes will be kept on-site to reduce potential downtime. If problem equipment is detected or should require service, the equipment will be returned and a qualified technician will perform the maintenance required. Use of the instrument will not be resumed until the problem is resolved. Backup instruments and equipment will be available on-site or within 1 day shipment to avoid delays in the field activities. Routine maintenance of field instruments will be documented in bound logbooks which will be kept with the field instrument. Spare parts and the maintenance schedule are presented on Table 11-1.

11.2 LABORATORY INSTRUMENT PREVENTATIVE MAINTENANCE

Preventative maintenance and periodic maintenance is performed as recommended by the manufacturers of the equipment in use in the laboratory. Spare parts are kept in inventory to allow for minor maintenance.

The laboratory staff performs preventive maintenance and repairs or coordinates with a vendor for the repair of all instruments. All laboratory instruments are maintained in accordance with manufacturer's specifications and the requirements of the specific method employed. This

maintenance is carried out on a regular, scheduled basis and is documented in the laboratory instrument service logbook for each instrument. Emergency repair or scheduled manufacturer's maintenance is provided under a repair and maintenance contract with factory representatives. The following Table 11-1 summarizes preventive maintenance schedules and critical spare parts inventories. Refer to the SOPs included in Attachment B for the preventative maintenance program for the ICP/MS and ICP.

11.3 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES

Inspection/acceptance requirements for laboratory supplies and consumables are documented in Section 3.10, Attachment A.

TABLE 11-1
REFINED METALS SITE
PREVENTATIVE MAINTENANCE PROCEDURES
SCHEDULE AND SPARE PARTS LIST

Instrument	Activity	Frequency	Spare Parts
ICP	Change peristaltic tubing	Every 8 hours	Tubing
	Change gas and instrument filters	As needed	Gases
	Check to make sure the gas supply is sufficient for days activities	Daily	
	Clean nebulizer	Daily	
Hot plates	Monitor temperature	Daily	
Ovens	Monitor temperature	Daily	
Refrigerators	Monitor Temperature	Daily	
OVA	Calibrate	Daily, throughout day	Batteries
	Check battery	Daily	Spare lamp
	Clean UV lamp, ion chamber and fan	When calibration fails or readings are erratic	
pH meter	Calibrate with two standard solutions	Daily, throughout day	pH buffers Electrodes
	Replace electrodes	As needed	Batteries
Conductivity Meters	Calibrate Check batteries	Daily Daily	Batteries
Thermometer	Check against calibrated thermometer	Yearly	

12.0 SPECIFIC ROUTINE PROCEDURES USED TO EVALUATE DATA PRECISION, ACCURACY AND COMPLETENESS

The purpose of this section is to indicate the methods by which it will be ensured that the data collected for this investigation falls in line with the DQOs for the site.

Factors considered in this assessment include, but are not limited to:

- The risk assessment parameters chosen based on conditions and possible receptors involved in the project.
- The contaminants known and/or suspected to be of concern on a project as they relate to the data quality level parameters chosen.
- The choice of analytical and sample preparation methods for contaminants of concern whose method detection limits will meet or exceed the data quality level concentrations for those contaminants.

Once these goals and objectives are evaluated and chosen, analytical data quality will be assessed to determine if the objectives have been met. In addition, the data will be reviewed for indications of interferences to results caused by sample matrices, cross contamination during sampling, cross contamination in the laboratory, and sample preservation and storage anomalies (i.e., sample holding time).

12.1 ACCURACY ASSESSMENT

Accuracy will be calculated on the average percent recovery of spiked samples. In order to assure the accuracy of the analytical procedures, an environmental sample shall be spiked with a known amount of the project target analytes. At a minimum, one spike sample shall be included in every

set of 20 samples analyzed on each instrument, for each sample matrix to be tested (i.e. soil, sediment and groundwater). The increase in concentration of the analyte observed in the spiked sample, due to the addition of a known quantity of the analyte, compared to the reported value of the same analyte in the unspiked sample determines the percent recovery. Accuracy is similarly assessed though determination of percent recoveries for laboratory control samples. Reference materials are essential to the evaluation of accuracy. Stock solutions for accuracy spikes and laboratory control samples shall be traceable to a source independent from the calibration standards. Accuracy is calculated using the equation below:

$$\%R = \frac{SSR - SR}{SA} x100 \quad \text{or} \quad \frac{SR}{TV} = 100$$

Where:

$\%R$ = percent recovery

SSR = spiked sample result

SR = sample result

SA = amount of spike

TV = true value (actual mass)

12.2 PRECISION ASSESSMENT

The precision of matrix spike and matrix spike duplicate, and field duplicate pairs or laboratory duplicate pairs will be expressed as relative percent difference (RPD) or relative standard deviation (RSD). RPD is derived from the absolute difference between duplicate analyses divided by the mean value of duplicates. The percent RSD is obtained by dividing the standard deviation by the average of the sample set. Equations for RPD and RSD are presented below:

$$RPD = \frac{|D_1 - D_2|}{((D1+D2)1/2)} \; x \; 100$$

Where:

D1 and *D2* = two replicate values

$$RSD = \frac{S}{X}; \; and S = [\frac{\sum_{i=1}^{n}}{}(x_i - \bar{x})^2 \; / \; (n-1)]^{1/2}$$

Where:

S = standard deviation

X = average of sample set

x_i = each observed value

x = the arithmetic mean of all observed values

n = total number of values

12.3 COMPLETENESS ASSESSMENT

Completeness is evaluated by dividing the total number of verifiable data points by the maximum number of data points possible and expressing the ratio as a percent. A usability criteria of 90 percent has been set for this project. Following completion of the analytical testing, the percent completeness will be calculated using the following equation:

$$Completeness(\%) = \frac{D}{Pxn} \; x \; 100$$

Where:

D = number of confident quantifications

P = number of analytical parameters per sample requested for analysis

n = number of samples requested for analysis

12.4 ASSESSMENT OF DATA

The field and laboratory data collected during this investigation will be used to evaluate groundwater flow and quality, characterize soil quality, confirm/determine concentrations of lead and cadmium within the buildings and determine whether past drainage areas have been affected by contaminant transport. The QC results associated with each analytical parameter for each matrix will be compared to the objectives presented in Sections 3.8 and 3.9 of this QAPP. Only data generated in association with QC results meeting these objectives will be considered usable for decision making purposes.

In addition, the data obtained will be both qualitatively and quantitatively assessed on a project-wide, matrix-specific, parameter specific and unit-specific basis. The assessment will be performed by the Quality Assurance Manager and the results will be presented and discussed in detail in the final investigation report. Factors to be considered in this assessment of the field and laboratory data will include, but not necessarily be limited to, the following:

- Were all samples collected using the methodologies and SOPs proposed in the QAPP?
- Were all proposed analyses performed in accordance with the SOPs provided in this QAPP?

- Were samples obtained from all proposed sampling locations and depths?
- Were samples received at the laboratory intact and within holding time requirements?
- Do any analytical results exhibit elevated detection limits due to matrix interferences or contaminants present at high concentrations?
- Were all data validated according to the validation documents proposed in this QAPP?
- Were any data found to be unusable (qualified as "R") based on the data validation results?
- Were any data found to be usable for limited purposes (qualified as "J") based on the data validation results?
- What affect due qualifiers applied as a result of data validation have on the ability to implement the project decision rules?
- Has sufficient data of appropriate quality been generated to support a human health and ecological screening risk assessment?
- Can valid conclusions be drawn for each area under this investigation or is further sampling required?
- Were all issues requiring corrective action fully resolved?
- Based on the overall findings of the investigation and this assessment, were the original project objectives appropriately defined? If not, have revised project objectives been developed?

13.0 CORRECTIVE ACTION

When field sampling activities or laboratory quality control results show the need for corrective action, immediate action will take place and will be properly documented. In the event that a problem arises, corrective action will be implemented. Any error or problem will be corrected by an appropriate action which may include:

- Replacing or repairing a faulty measurement system;
- Discarding erroneous data;
- Collecting new data; and
- Accepting the data and acknowledging a level of uncertainty.

13.1 FIELD CORRECTIVE ACTION

The PI will be responsible for all field quality assurance. Any out of protocol occurrence discovered during field sampling will be documented in the field notebook and immediate corrective action will be taken. For problems or situations which cannot be solved through immediate corrective action, the PI will immediately notify the Contractor's Project Manager. The AGC Project Manager and PI will investigate the situation and determine who will be responsible for implementing the corrective action. Corrective action will be implemented upon approval by the AGC Project Manger. The AGC Project Manager will verify that the corrective action has been taken, appears effective, and at a later date, verify that the problem has been resolved. The successfully implemented corrective action will be documented in the field logbook by the PI. Any deviations from the quality assurance protocol in the QAPP must be justified, approved by the AGC Project Manager (and the USEPA, if necessary), and properly documented.

13.2 LABORATORY CORRECTIVE ACTION

Corrective action will be implemented to correct discrepancies found which affect the validity or quality of analytical data and to identify any analytical data that may have been affected. Limits of data acceptability for each parameter and sample matrix are addressed in the instrument manuals, USEPA Methods and/or Laboratory QA Manual (Attachment A). Whenever possible, immediate corrective action procedures will be employed. All analyst corrective actions are to be followed according to the instrument manuals, USEPA Methods, or Laboratory QA Manual. Any corrective action performed by analyst will be noted in laboratory logbooks.

Laboratory personnel noting a situation or problem which cannot be solved through immediate corrective action, will notify the Laboratory QA Supervisor. The QA Supervisor will investigate the extent of the problem and its effect on the analytical data generated while the deficiency existed. All data suspected to be affected will be scrutinized to determine the impact of the problem on the quality of the data. If it is determined that the deficiency had no impact on the data, this finding will be documented. If the quality of the analytical data were affected, the Laboratory Program Manager and Contractor's Project Manager will be notified immediately so that courses of action may be identified to determine how to rectify the situation.

The laboratory must take corrective action if any of the quality control data generated during the laboratory analyses are outside the method criteria. Corrective action for out-of-control calibrations is to recalibrate the instrument and reanalyze the samples. A sequence is specified in the USEPA specified methods when problems in analyses are encountered. The laboratory will follow these procedures exactly and document the problems encountered and corrective action in a case narrative enclosed with each data deliverables package.

The Laboratory QA Supervisor will be responsible for informing the Laboratory Program Manager and Sampling Contractor's Project Manager the effects on the data, the data affected and the corrective action taken. It is also the Laboratory QA Supervisor's responsibility to verify the corrective action was performed, appears effective, and at a later date, the problem was resolved.

Documentation of corrective actions taken by laboratory are outlined in Section 4, Attachment A. Reports will be completed to document nonconformances and the corrective actions taken. Copies of nonconformance reports will be included as part of the laboratory deliverable for this project.

13.3 CORRECTIVE ACTION DURING DATA VALIDATION AND DATA ASSESSMENT

Upon completion, sample data packages will be sent from the laboratory to the AGC QA Manager for data validation. If all project samples are not present in the data packages or any deficiencies affecting the sample results are noted, the QA Manager will contact the Laboratory Program Manager. The Laboratory Program Manager will consult with the Laboratory QA Supervisor and respond in writing to any inquiries and provide any changes to the data packages to the QA Manager. Any errors, problems, questionable data values, or data values outside established control limits will be corrected by the appropriate action which may include disregarding erroneous data, collecting new data, and accepting the data and acknowledging a level of uncertainty. The data validation report will provide a description of the usability of the data.

14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

14.1 CONTENTS OF PROJECT QA REPORTS

After project initiation, the AGC Project Manager, in conjunction with the Quality Assurance Manager, will submit with the monthly progress report a written quality assurance summary section in which data quality information collected during the task is summarized. The report will be provided to the RMC Project Manager, who will, in turn after review, forward it to the EPA Remedial Project Manager. The quality assurance summary will provide information on the performance of measurement systems and data quality and will contain at a minimum, the following information (when applicable):

- A description of the actions which have been taken during the sampling event including but not limited to data collection and implementations of the QAPP;
- The results of sampling and tests and all other data received or generated;
- The status and coverage of various laboratory and field quality assurance project activities, including any delays realized or anticipated to affect the project schedule;
- Data quality assurance reviews including assessment of accuracy, precision, completeness, representativeness and comparability;
- Any significant field observations noted in the field notebook during sampling procedures;
- Changes in key personnel and updates on any training provided;
- Significant quality assurance problems encountered, corrective actions taken, progress and improvements, plans and recommendations for further implementation or updating of the QAPP; and

- A summary of the results of laboratory and field performance and system audits, if conducted.

Sample analysis results will be submitted to the RMC Project Manager as they become available, following QA/QC review, for inclusion in the Progress Report. A tabulation of analytical data, including detection limits and data flags will be included in a data validation report. The data validation report will also include a summary of the qualitative and quantitative reliability of the analytical data.

14.2 FREQUENCY OF REPORTS

The QA Report will be provided as a section in the monthly progress reports to the RMC Project Manager who will be responsible for submitting updates to the EPA and IDEM. Reporting will begin at the end of the first full month following the lodging date of the Consent Decree, and continuing throughout the period the Consent Decree is effective. The data validation reports will be submitted to the RMC Project Manager as they become available.

14.3 INDIVIDUALS RECEIVING/REVIEWING QA REPORTS

All individuals identified in the Project Organization Chart (excluding the Laboratory personnel and the field staff) will receive copies of the monthly QA Report.

ATTACHMENT A

OF THE

QUALITY ASSURANCE PROJECT PLAN

REFINED METALS CORPORATION SITE

LABORATORY QUALITY ASSURANCE MANUAL

AND

STATEMENT OF QUALIFICATIONS

1.0 TITLE PAGE

TriMatrix Laboratories, Inc.

QUALITY ASSURANCE MANUAL

Revision Number: 2.1

Effective Date: 10/97

Approvals:

Quality Assurance Manager: _____ Date: 10/17/97

Laboratory Manager: _____ Date: 10/17/97

TABLE OF CONTENTS

TABLE OF CONTENTS

(con't)

TABLE OF CONTENTS

(con't)

Page #

TABLE OF CONTENTS

(con't)

Page #

TABLE OF CONTENTS
(con't)

Page #

TABLE OF CONTENTS
(con't)

Page #

TABLE OF CONTENTS
(con't)

TABLE OF CONTENTS
(con't)

Page #

FIGURES

FIGURES
(con't)

TABLES

APPENDICES

3.0 QUALITY SYSTEM

3.1 INTRODUCTION: THE *TriMatrix* QUALITY SYSTEM

3.1.1 The Purpose of This Manual

The purpose of this manual is to outline the organization, specify the procedures, and define the technical requirements to be utilized by TriMatrix Laboratories, Inc. The goal is to ensure that the data is of the required quality, is reproducible, and is generated in a time-efficient manner. This manual details a Quality Assurance/Quality Control (QA/QC) program affecting every individual involved in the analytical efforts at TriMatrix, from project initiation, to sample receiving, to final report generation. Some of the areas covered will only have a cursory discussion, while others will be covered in detail, or will be included in more than one section. This manual describes the realistic functioning of the laboratory and the quality programs in place, understanding that not every situation is covered nor every contingency explored.

3.1.2 The Need for Analytical Quality Assurance/Quality Control

In the increasingly competitive business of environmental laboratory services, the primary tenet of continued success is to efficiently provide results of the necessary quality. TriMatrix agrees with this principle, considers analytical quality assurance and quality control to be of great importance, and has incorporated it as a central pillar of our efforts to remain on the leading edge of this field. The requirements we place on ourselves are in concert with the needs and agendas of other organizations, such as the Environmental Protection Agency (EPA), our governmental and industrial clients, and the various state and local regulatory agencies.

Quality assurance and quality control absorb nearly fifty percent of all the available effort for routine analysis and are continuing to evolve and grow in importance. It would seem that this quality control effort would detract from profitability and not be practical for a commercial laboratory. However, the

quality assurance activities are absolutely essential for two reasons: 1) accurate analytical data can be obtained only with the concurrent use of extensive quality control to monitor the many process variables that can introduce errors into chemical analyses; and 2) clients make crucial business decisions based on the data supplied by the laboratory regarding the concentration of analytes in water, effluent, soil, air, and waste samples. Lab data which is not properly supported by quality assurance/quality control can be questionable and lead to faulty or erroneous decisions in field situations. In the total analytical effort, the space taken by quality control samples in each testing batch ensures the accuracy of the client analyses.

3.1.3 Definition of Terms

3.1.3.1 Quality Assurance

Quality Assurance (QA) is defined as those operations and procedures which are undertaken to provide measurement data of defined quality that have a stated probability of being correct. The measurement system that is part of the quality assurance program, must be in a state of statistical control in order to justify the probability statement.

The operations and procedures that are established as part of the overall quality assurance program encompass all aspects of the laboratory operations including but not limited to: organizational structure, human resources, physical resources, metrology, methodology, data reduction and validation, and trouble shooting. All aspects of QA are organized, implemented, and monitored through written operating procedures.

3.1.3.2 Quality Control

Quality control is defined as the basic ingredients necessary to produce a good measurement program. These ingredients include but are not limited to: correct methodologies and proper use of these

methodologies; proper calibration and calibration verification; statistical monitoring of accuracy and precision; interference monitoring; reagent control; data processing; analyst training and certification; and instrument maintenance.

These basic ingredients must be accompanied by proper documentation. Adequate records are maintained to support our claims for the quality of data, to identify assignable causes in the event of measurement problems, to improve the accuracy and precision of our measurement systems, and to provide a historical record of our testing activities.

3.1.3.3 Quality Assessment

Quality assessment is defined as those specific steps which are utilized to assess the quality of the measurement process. These steps include but are not limited to: use of control charts to plot multiple data points versus time and verify whether the testing is in a state of statistical control; quality control samples (e.g. standards, laboratory control samples, blanks, duplicates, spikes, etc.); internal performance audits (system, documentation, surveillance); external performance audits (auditors from clients and regulatory agencies); certification programs conducted by states such as Michigan, Wisconsin, and California; annual performance evaluation sample programs from the USEPA (WS, WP, DMR-QA), state certifying agencies, and certain clients.

3.2 QUALITY POLICY STATEMENTS FROM MANAGEMENT

As communicated from top management down through the entire organization, TriMatrix Laboratories, Inc. is driven by the following quality objectives and quality commitments:

3.2.1 Corporate Quality Objectives

- To create and maintain a uniform and controlled pattern for the performing of every routine task within the organization, based on standard operating procedures.

- To generate laboratory data which is scientifically sound, legally defensible, and of known high quality.

- To build the necessary quality into our work place and services to ensure successful relationships with, and contribute to the future of, our customers, employees, and vendors.

- To develop, deliver, and maintain excellence in every area of our operations.

- To work with customers to provide services that always meet or exceed their expectations.

3.2.2 Corporate Quality Commitments

- To support quality and continually underwrite the substantial cost of quality assurance even though these expenses do not result in increased productivity or a tangible product.

- To maintain a work environment in which employees are free from commercial pressures in the performance of their duties.

- To maintain a work environment in which all employees are free from organization or client related pressures that may influence the quality of their work.

- To ensure that client confidentiality and information are strictly protected.

- To implement continuous improvement in every area of laboratory activity.

- To create and maintain a Quality Culture where there is an all-encompassing determination to meet the needs of customers, and deliver quality to the customer.

Included with these improvements and commitments is an annual review process where the management of TriMatrix Laboratories performs a comprehensive review of all quality systems in place. This review is performed to monitor the effectiveness of the quality system and provide feedback for continuous improvement. Because this QA manual plays an important role in the TriMatrix quality system, it will be a central part of the annual review by the management of TriMatrix Laboratories.

3.3 ORGANIZATION AND RESPONSIBILITIES

An efficiently-operating organization requires a quality control program which facilitates a high level of communication and information flow from all directions. To implement this objective, each person in our organization gives input to, and receives information from, the quality system. This information flow allows directives from management to be implemented with minimum disruption, permits analysts to measure the caliber of their work, and provides the means for creating improvements.

3.3.1 Corporate Structure

Flow of both administrative and quality control information is presented in Figure 1. The purpose of this diagram is to graphically display the corporate philosophy concerning the interaction of QA/QC and the generation of analytical data. The general flow of data in this format gives QA/QC independence in fulfilling its functions while still acting as a liaison in coordination with the administrative chain. To further explain this interaction, a more detailed description of roles and responsibilities are presented for each key laboratory position.

3.3.2 Laboratory Manager

The responsibilities of the Laboratory Manager are directed at the overall operation and management of all corporate laboratories. Primary responsibilities include, but are not limited to: 1) develop and meet budgets established for the laboratory, 2) manage analytical services productivity and quality, 3) oversee and develop new business activities including client relations development, 4) plan analytical services organization, leadership and management programs, and 5) develop and manage human resources including career path planning.

3.3.3 Quality Assurance Manager

The Quality Assurance Manager is primarily responsible for the implementation, maintenance, reporting and development of all QA/QC activities performed within the laboratory. Some of the duties performed include, but are not limited to: 1) QA/QC systems development and monitoring, 2) coordination of all documentation procedures including the development and control of standard operation procedures, 3) monitoring method and quality control requirements as published by regulatory agencies, 4) performing internal lab audits including the submission of double-blind evaluations to the analytical system, 5) maintenance of in-house QA/QC monitoring procedures and policies, 6) providing quality assurance training to all staff members, 7) managing all activities of other QA/QC staff members, 8) performing duties as Deputy Technical Director when necessary.

3.3.4 Technical Director

The Technical Director is responsible for the overall technical capabilities and direction of the laboratory. Specific responsibilities include: 1) organization and management of all new analytical technologies developed by the laboratory, 2) preparation and publication of all laboratory Quality Assurance Project Plans (QAPPs), 3) equipment procurement management.

3.3.5 Health and Safety Officer

The Health and Safety Officer is responsible for the implementation, monitoring, and maintenance of all laboratory safety and chemical hygiene programs. Specific responsibilities include: 1) development and maintenance of health and safety programs and manuals and, 2) management and development of the health and safety coordinator.

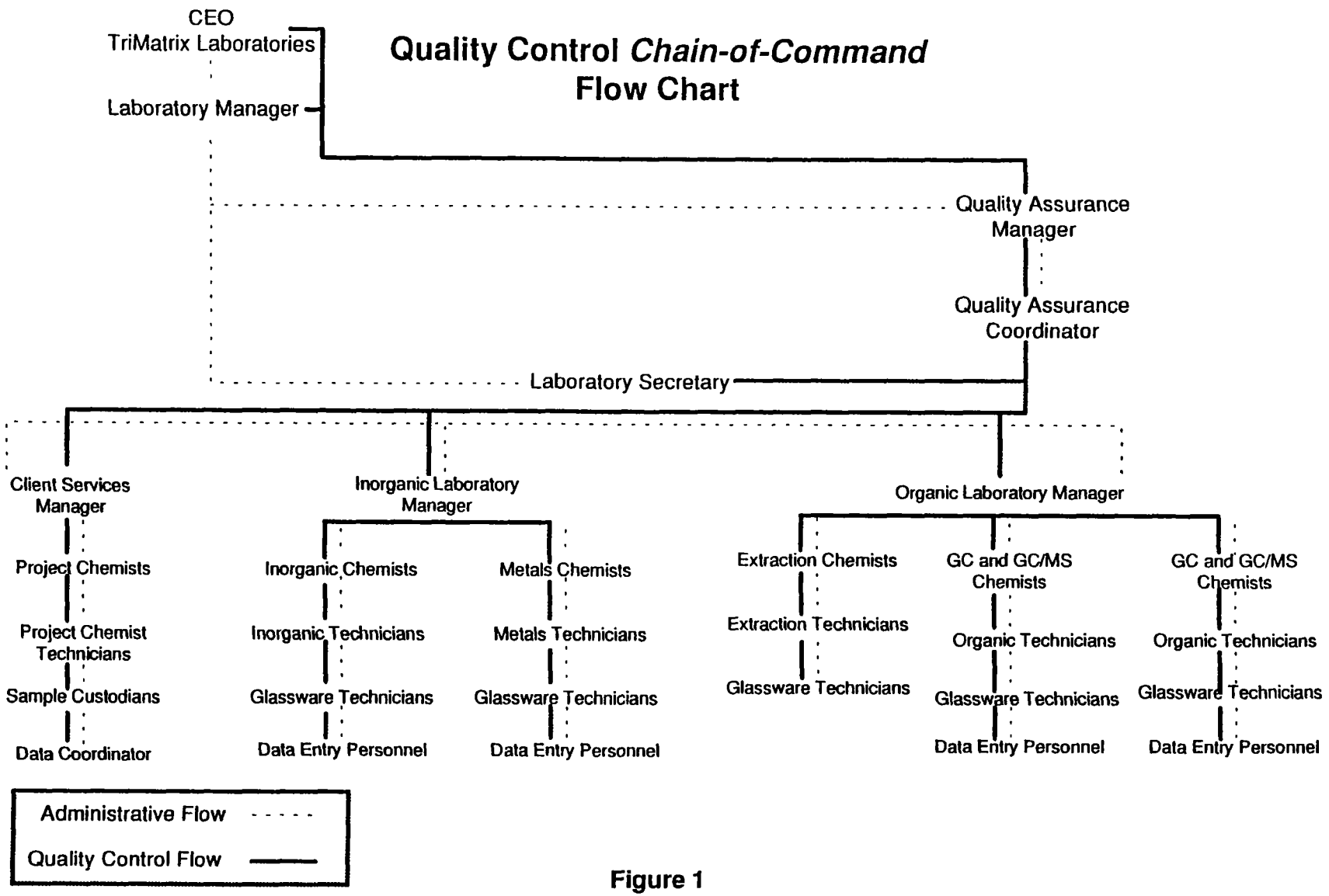

Quality Control *Chain-of-Command* Flow Chart
CEO
TriMatrix Laboratories
Laboratory Manager
Quality Assurance Manager
Quality Assurance Coordinator
Laboratory Secretary
Client Services Manager
Project Chemists
Project Chemist Technicians
Sample Custodians
Data Coordinator
Inorganic Laboratory Manager
Inorganic Chemists
Inorganic Technicians
Glassware Technicians
Data Entry Personnel
Metals Chemists
Metals Technicians
Glassware Technicians
Data Entry Personnel
Organic Laboratory Manager
Extraction Chemists
Extraction Technicians
Glassware Technicians
GC and GC/MS Chemists
Organic Technicians
Glassware Technicians
Data Entry Personnel
GC and GC/MS Chemists
Organic Technicians
Glassware Technicians
Data Entry Personnel
Administrative Flow
Quality Control Flow

Figure 1

3.3.6 Laboratory Area Managers

Laboratory Area Managers are responsible for the overall supervision of their respective laboratory sections or areas. General responsibilities include management of staff activities, such as scheduling and method development; budgeting; training; and general supervision. Each section or Area Manager is also responsible for all data generated and all correspondence produced by their respective staff members.

3.3.6.1 Client Services

The laboratory Client Services Manager supervises both the client services group and the administrative staff within the laboratory. Responsibilities of the Client Services Manager includes those outlined in section 3.3.6 with specific emphasis on the following activities: 1) development and management of all project chemists, project chemist technicians, log-in staff, bottle preparation staff, and laboratory administrative staff; 2) project management; 3) coordination of proposal preparation and marketing activities for existing and new clients; 4) monitoring of final report turnaround times and; 5) maintenance of client satisfaction with laboratory services.

3.3.6.2 Inorganic Laboratory (Metals and Non-metals)

The Inorganic Laboratory Manager supervises all aspects of the inorganic laboratory, which includes the metals and non-metals laboratories. Responsibilities include those outlined in section 3.3.6 with specific emphasis on the following activities: 1) development and management of all inorganic chemists, analysts, technicians, and data entry personnel, 2) implementation of quality systems and controls within the inorganic laboratory, 3) scheduling of all inorganic testing activities, 4) meeting productivity goals and project deadlines, 5) technical development of the inorganic laboratory staff, 6)

development and maintenance of the inorganic laboratory's SOPs, 7) coordination of methods development with the inorganic staff and Technical Director, 8) approval of all inorganic laboratory data, or the delegation thereof, 9) monitoring of all procurement activities and, 10) reporting to management on overall inorganic laboratory performance.

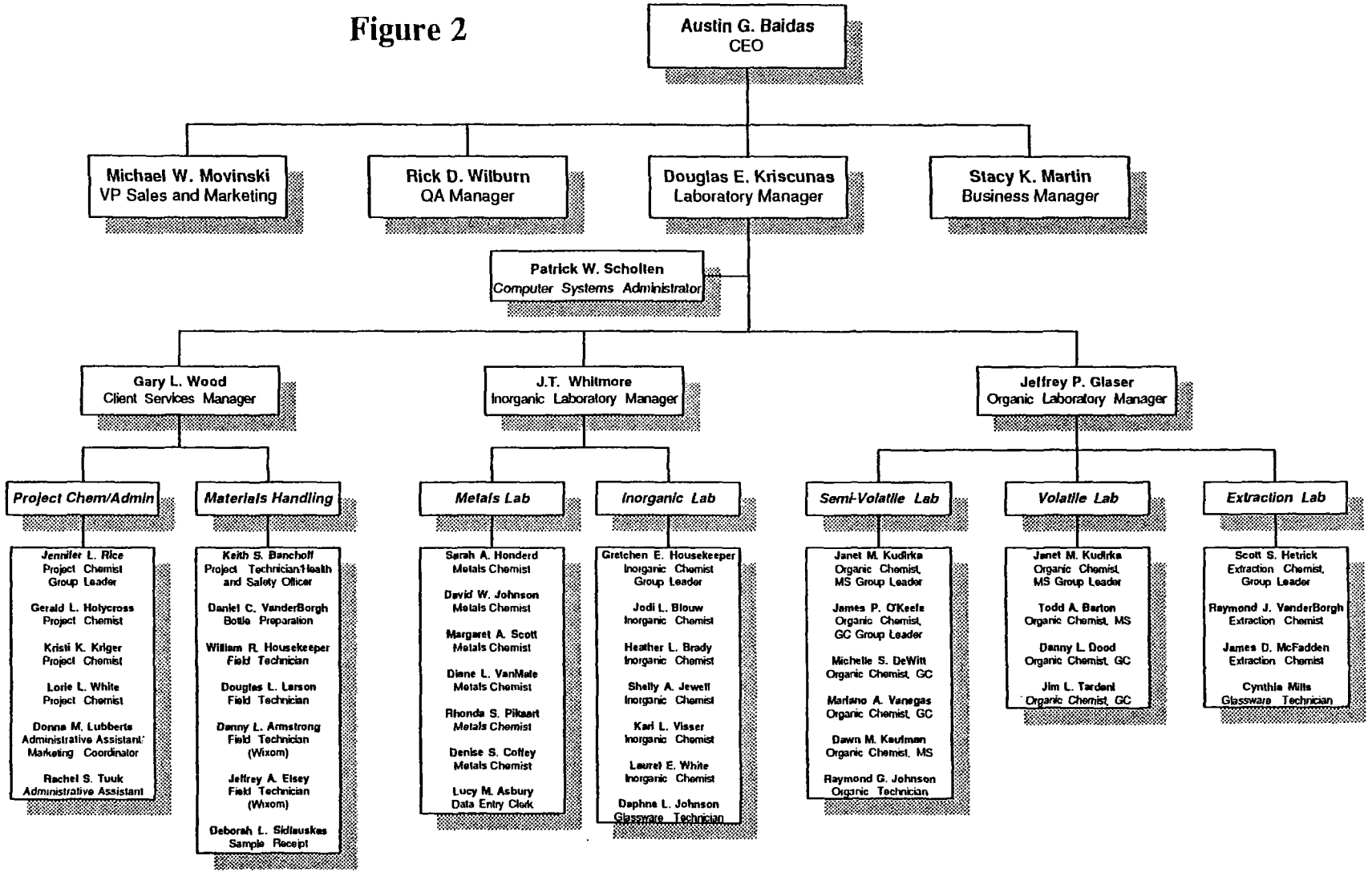
3.3.6.3 Organic Laboratory (Semi-Volatile and Volatile)

The Organic Laboratory Manager supervises all aspects of the volatile organics and semi-volatile organics laboratories. Responsibilities include those outlined in section 3.3.6 with specific emphasis on the following activities: 1) development and management of all organic chemists, analysts, technicians, and data entry personnel, 2) implementation of quality systems and controls within the organic laboratories, 3) scheduling of all organic extraction and testing activities, 4) meeting productivity goals and project deadlines, 5) technical development of the organic laboratories' staff, 6) development and maintenance of the organic laboratory SOPs, 7) coordination of methods development with organic staff members and the Technical Director, 8) approval of all organic data, or the delegation thereof, 9) monitoring of all procurement activities and, 10) reporting to management on overall performance of the organic laboratories.

3.3.7 Organizational Chart

Presented in Figure 2 is an organizational chart illustrating the structure within the laboratory.

Figure 2


Austin G. Baidas
CEO
Michael W. Movinski
VP Sales and Marketing
Rick D. Wilburn
QA Manager
Douglas E. Kriscunas
Laboratory Manager
Stacy K. Martin
Business Manager
Patrick W. Scholten
Computer Systems Administrator
Gary L. Wood
Client Services Manager
J.T. Whitmore
Inorganic Laboratory Manager
Jeffrey P. Glaser
Organic Laboratory Manager
Project Chem/Admin
Materials Handling
Metals Lab
Inorganic Lab
Semi-Volatile Lab
Volatile Lab
Extraction Lab
Jennifer L. Rice
Project Chemist
Group Leader
Gerald L. Holycross
Project Chemist
Kristi K. Kriger
Project Chemist
Lorie L. White
Project Chemist
Donna M. Lubberts
Administrative Assistant/
Marketing Coordinator
Rachel S. Tuuk
Administrative Assistant
Keith S. Banchoff
Project Technician/Health
and Safety Officer
Daniel C. VanderBorgh
Bottle Preparation
William R. Housekeeper
Field Technician
Douglas L. Larson
Field Technician
Danny L. Armstrong
Field Technician
(Wixom)
Jeffrey A. Elsey
Field Technician
(Wixom)
Deborah L. Sidlauskas
Sample Receipt
Sarah A. Honderd
Metals Chemist
David W. Johnson
Metals Chemist
Margaret A. Scott
Metals Chemist
Diane L. VanMale
Metals Chemist
Rhonda S. Pikaart
Metals Chemist
Denise S. Coffey
Metals Chemist
Lucy M. Asbury
Data Entry Clerk
Gretchen E. Housekeeper
Inorganic Chemist
Group Leader
Jodi L. Blouw
Inorganic Chemist
Heather L. Brady
Inorganic Chemist
Shelly A. Jewell
Inorganic Chemist
Karl L. Visser
Inorganic Chemist
Laurel E. White
Inorganic Chemist
Daphne L. Johnson
Glassware Technician
Janet M. Kudirka
Organic Chemist,
MS Group Leader
James P. O'Keefe
Organic Chemist,
GC Group Leader
Michelle S. DeWitt
Organic Chemist, GC
Mariano A. Venegas
Organic Chemist, GC
Dawn M. Kaufman
Organic Chemist, MS
Raymond G. Johnson
Organic Technician
Janet M. Kudirka
Organic Chemist,
MS Group Leader
Todd A. Barton
Organic Chemist, MS
Danny L. Dood
Organic Chemist, GC
Jim L. Tardent
Organic Chemist, GC
Scott S. Hetrick
Extraction Chemist,
Group Leader
Raymond J. VanderBorgh
Extraction Chemist
James D. McFadden
Extraction Chemist
Cynthia Mills
Glassware Technician

3.3.8 Deputy Technical Director/Deputy Quality Assurance Officer

In the absence of the Laboratory Technical Director, the Quality Assurance Manager is designated as the Deputy Technical Director with the responsibility of fulfilling an interim role as outlined in sections 3.3.4 and 3.5.1.2 of this manual.

In the absence of the Quality Assurance Manager, the QA/QC Coordinator is designated as the Deputy Quality Assurance Officer with the responsibility of fulfilling an interim role as outlined in sections 3.3.3 and 3.5.1.3 of the manual.

3.4 RELATIONSHIPS

Relationships within the analytical laboratory, in concert with management, are organized into three main categories: Technical Operations, Support Services, and the Laboratory Quality System. The relationships between management and these operational services and systems must be fully defined and monitored in order to maintain the delicate balance in a cost-effective, highly-technical, quality laboratory operation. An overview of each of these relations is presented below:

3.4.1 Management-Technical Operations

The relationship between management and technical operations is illustrated in Figure 3. In this relationship, the main role of management is to provide guidance and financial support to the programs and directives of the Technical Director. The organization, under the direction of the CEO and Laboratory Manager, is composed of the Deputy Director and all Area Managers. Through this structure, technical operational enhancements and developments occur and are applied through the laboratory staff.

3.4.2 Management-Support Services

The relationship between management and all branches of support services is illustrated in Figure 4. In this relationship, management's role is substantial in the day-to-day operation of these support groups.

RELATIONSHIPS
MANAGEMENT - TECHNICAL SERVICES

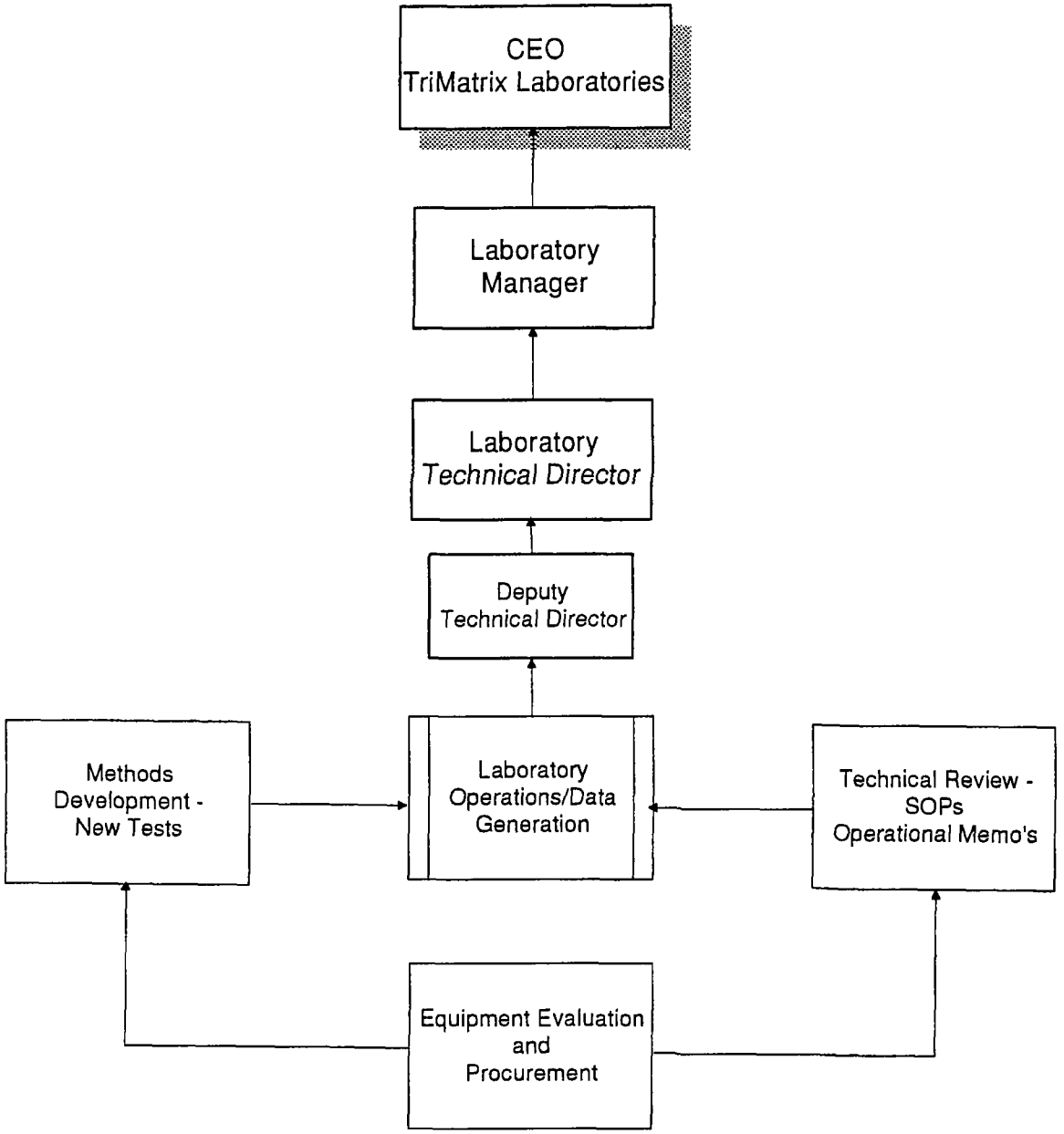


Figure 3

RELATIONSHIPS

MANAGEMENT - SUPPORT SERVICES

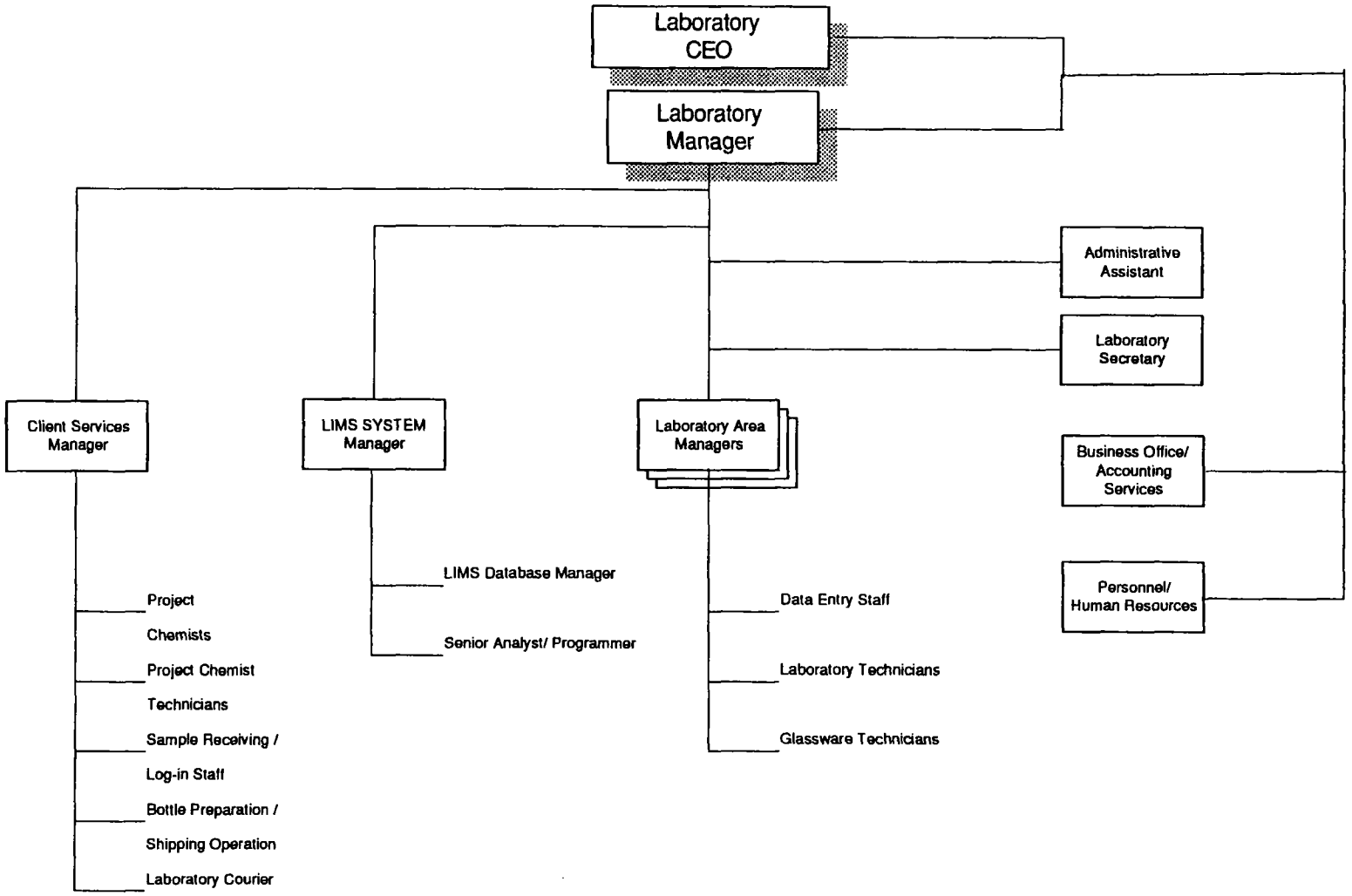


Figure 4

The primary laboratory support groups are Client Services, Marketing and Sales, and LIMS system support. These groups report directly to the Laboratory Manager for all aspects of their daily activities.

Secondary relationships are maintained with the Laboratory Administrative Assistant, Secretary and Accounting and Human Resources Departments. Some groups within this secondary category maintain relationships not only with the Laboratory Manager, but other management groups within the TriMatrix organization.

A tertiary relationship has been developed between the Laboratory Manager and the laboratory Area Managers. This relationship supports such activities as productivity monitoring, cost containment, equipment procurement, operations management, personnel/human resources activities, technical support, data validation, and method development.

3.4.3 Management-Quality System

The relationship between management and the laboratory quality system is illustrated in Figure 5. In this relationship, management plays a secondary role in the overall scheme. This secondary role is designed to provide the corporate quality assurance officer with guidance, corporate perspective, and structured support in the development, implementation, and maintenance of quality system programs and activities.

This relationship is vital to the success of TriMatrix Laboratories. Without a good cost-effective quality system, the overall caliber of laboratory data and the success of all laboratory operations would be jeopardized.

A tertiary relationship also exists between management, the quality system, and the supporting cast of the laboratory QA/QC staff. This relationship includes but is not limited to: laboratory management directives, human resources/personnel activities, and corporate directives. These activities are implemented and maintained without disruption to the quality system.

RELATIONSHIPS
MANAGEMENT - Quality System

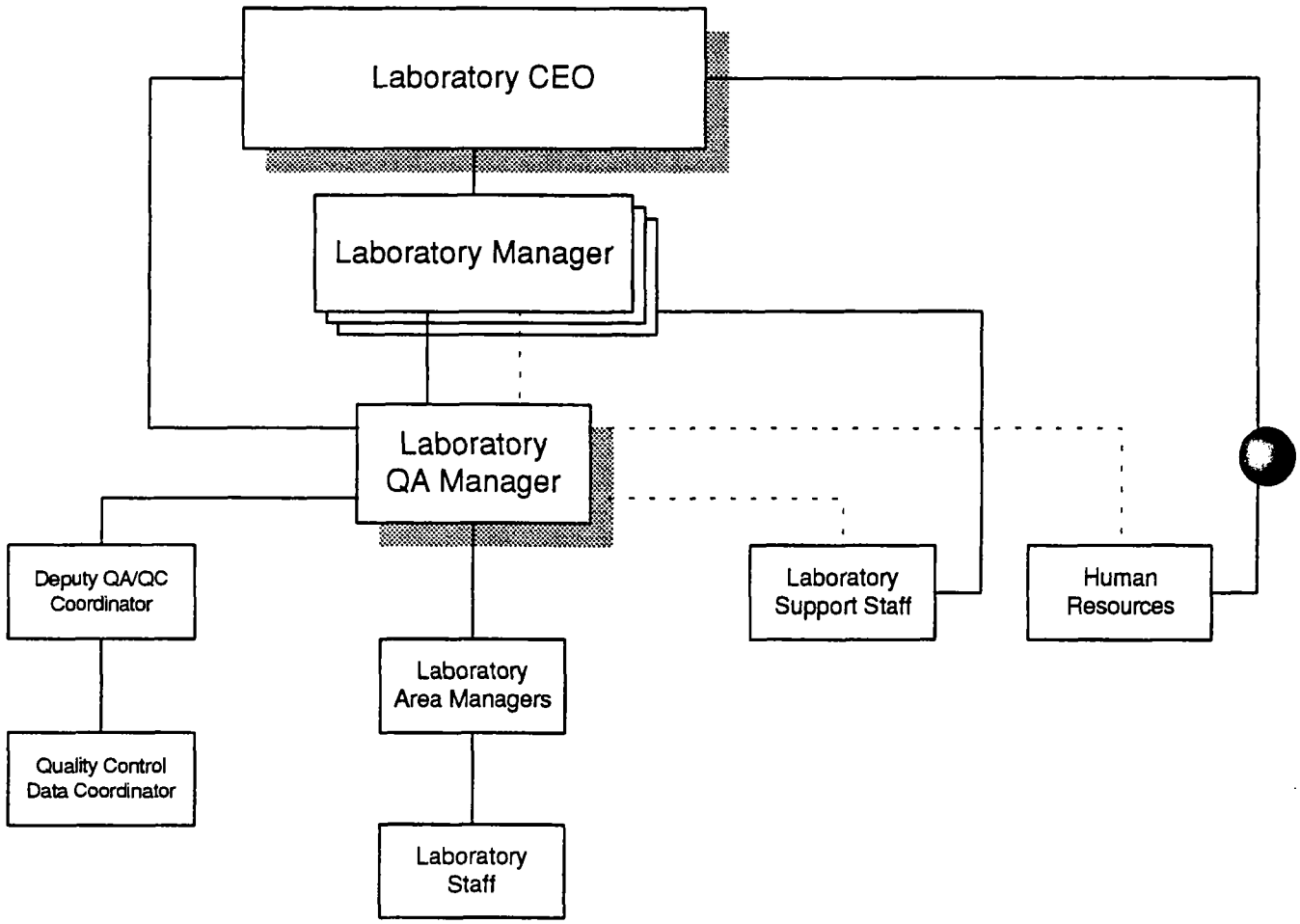


Figure 5

3.5 JOB DESCRIPTIONS

3.5.1 Management Staff Members

3.5.1.1 Laboratory Manager

Job Description

The Laboratory Manager (LM) directs the laboratory. The LM works through the area managers and their staff to improve data quality, overall productivity, staff development, safety/training programs, and overall profitability. This position has profit/loss accountability. Budgets are developed with senior management annually. The LM is also involved in business development/sales activities directly, as well as having sales staff reporting to him.

Background/Educational Requirements

The LM possesses minimally a bachelors degree in science, preferably chemistry. Advanced degrees in the sciences and/or business are preferred. The LM has a minimum of 10 years direct work experience in the environmental testing industry. This work experience includes having conducted environmental analyses and several years of demonstrated supervisory experience.

Duties and Responsibilities

1. Development and fulfillment of budgets.
2. Management of division productivity and quality.
3. Management of proposal preparation.
4. Development of new business and maintenance of client relationships.
5. Development of laboratory organization, leadership and management planning.
6. Working with the Human Resources department to develop staff members and their career paths.
7. Perform all other duties as deemed necessary by management.

3.5.1.2 Technical Director Laboratory Operations

Job Description

The Technical Director (TD) of laboratory operations is responsible for the development and improvement of all technical operations within the laboratory division. The TD oversees the investigation of all new instruments and apparatuses, method development, and general technical advancement of the laboratory. The Technical Director is also responsible for informing the deputy Technical Director of all current and pending projects and activities.

Duties and Responsibilities

1. The continual technical development of the TriMatrix laboratory pertaining to current and future analytical practices.
2. Overseeing the technical development of all TriMatrix employees in the area of USEPA method comprehension and implementation.
3. Development of new analytical methods within the laboratory.
4. Providing technical advice regarding all equipment and apparatus procurement and acquisitions.
5. Performing technical review of all Quality Assurance Project Plans (QAPPs).
6. Perform all other duties as deemed necessary by management.

3.5.1.3 Quality Assurance Manager

Job Description

The Quality Assurance (QA) Manager has the responsibility for development, implementation, improvement, and maintenance of all quality systems within the TriMatrix laboratory. The QA Manager monitors all analytical methods and procedures performed by the laboratory, and provides assurance that they comply with regulatory agency requirements.

Background Education Requirements

The Manager possesses a B.S. in science, preferably chemistry, and suitable work experience. The work experience includes several years of analytical work and a demonstrated ability to work with and train staff members. A strong working knowledge of quality assurance and statistical quality control procedures, specifically as they apply to USEPA analytical protocols, is required.

Duties and Responsibilities

1. Development and implementation of systems that will measure and monitor the quality of all laboratory data.
2. Maintenance of the documentation system for generation, control, and archiving of all laboratory forms, SOPs, and protocols.
3. Approving all SOPs and monitoring their compliance with regulatory agency requirements.
4. Maintaining and updating the laboratory Quality Assurance Manual.
5. Submitting a monthly report identifying the quality control data generated by each lab area, the percent of data in control, and what activities were undertaken for out-of-control data.
6. Continually investigating and improving procedures to increase the percent of data within control limits.
7. Maintenance of federal, state, and industrial certifications and accreditations, as required.
8. Monitoring internal quality programs within the laboratory and reporting their status to management.
9. Training and documenting all new staff members in all aspects of the laboratory quality system.
10. Perform all other duties as deemed necessary by management.

3.5.1.4 Client Services Manager

Job Description

The Client Services Manager has the responsibility for the general supervision of the project chemist, project technicians, sample

receiving staff, bottle preparation staff, and laboratory administrative staff in a professional manner. These oversight responsibilities include meeting project due dates, preparing and reviewing quotations, project initiation and management, client satisfaction management, and the supervision and training of staff. The Client Services Manager strives for improvement in the acquisition and on-time delivery of laboratory projects.

Background/Educational Requirements

The Manager possesses a B.S. in science, preferably chemistry, and has 5-10 years of work experience. The work experience includes 3-5 years of laboratory experience, involvement in client management activities, and a demonstrated ability to supervise and train laboratory staff.

Duties and Responsibilities

1. Responsible for the productivity and quality of the client services group.
2. Management of large level 3 or higher projects.
3. Quality control program implementation and maintenance.
4. Supervision and technical development of employees.
5. Development and maintenance of standard operating procedures.
6. Assisting and coordinating marketing activities through proposal preparation and client visitation.
7. Perform all other duties as deemed necessary by management.

3.5.1.5 Organic Laboratory Manager

Job Description

The Organic Laboratory Manager has the responsibility of overseeing the supervision and operation of the volatile and semi-volatile organic laboratories in a professional manner. These oversight responsibilities include meeting project schedules and the supervision

and training of staff members. The Organic Manager will continually work to improve the quality of data generated.

Background/Educational Requirements

The Manager possesses a B.S. degree in science, preferably chemistry, and 5-10 years work experience. The work experience includes a minimum of 5 years performing organic analyses utilizing GC and GC/MS techniques. Experience must also include time spent in the extraction laboratory performing extractions of samples for semi-volatile analyses. The Manager must also demonstrate the ability to supervise and train organic staff members.

Duties and Responsibilities

1. Responsible for the productivity and quality of organic chemistry projects and data.
2. Operation and maintenance of instrumentation and apparatus.
3. Quality control program implementation and maintenance.
4. Reviewing and final approval of all organic data.
5. Developing schedules for in-house work that will allow on-time report generation.
6. Supervision of supply acquisition activities.
7. Supervision and technical development of employees.
8. Development and maintenance of standard operating procedures.
9. Methods development.
10. Perform all other duties as deemed necessary by management.

3.5.1.6 Inorganic Laboratory Manager

Job Description

The Inorganic Laboratory Manager has the responsibility of overseeing the supervision and operation of the metals and non-metals laboratories in a professional manner. These oversight responsibilities include meeting project schedules and the supervision

and training of staff. The Inorganic Laboratory Manager continually works to improve the quality of data generated.

Background/Educational Requirements

The Manager possesses a B.S. in science, preferably chemistry, and suitable work experience. The work experience includes 5-10 years of performing metals and inorganic wet chemistry analysis, and a demonstrated ability to supervise and train staff members.

Duties and Responsibilities

1. Responsible for the productivity and quality of inorganic chemistry projects and data.
2. Assuring proper operation and maintenance of instrumentation.
3. Quality control program implementation and improvement.
4. Reviewing and performing approval of all inorganic laboratory data.
5. Developing schedules for in-house work that will allow on-time report generation.
6. Supervision of supply acquisition activities.
7. Supervision and technical development of employees.
8. Development and maintenance of standard operation procedures.
9. Methods development.
10. Perform all other duties as deemed necessary by management.

3.5.2 Technical Staff Members

3.5.2.1 Organic MS Group Leader

Job Description

The Organic MS Group Leader performs organic analyses on environmental samples utilizing GC/MS. The Organic MS Group Leader is responsible for conducting analyses, troubleshooting instrumentation, performing the required quality control activities, and scheduling analyses so that projects will be completed on-time. The

implementation of new methods will be required as new environmental regulations mandate or allow their use.

Background/Educational Requirements

The Organic MS Group Leader will possess a science degree, preferably in chemistry, and/or suitable work experience. The work experience will include several years of performing organic analyses and a demonstrated ability to supervise and train organic staff members.

Duties and Responsibilities

1. Responsible for the productivity and quality of organic chemistry projects and data.
2. Operation, maintenance, and troubleshooting of instrumentation.
3. Quality control program implementation and maintenance.
4. Reviewing organic data.
5. Developing schedules for in-house work that will allow on-time report generation.
6. Maintenance of acceptable productivity.
7. Supervision and technical development of employees.
8. Improving all aspects of operations while emphasizing quality and efficiency.
9. Developing and researching new and better procedures.
10. Performing analyst and method certification as needed.
11. Maintaining a complete lab notebook in an organized manner.
12. Adhering to lab safety procedures.
13. Keeping lab neat and clean.
14. Perform all other duties as deemed necessary by management.

3.5.2.2 Organic GC Group Leader

Job Description

The Organic GC Group Leader performs organic analyses on environmental samples utilizing GC and High Performance Liquid

Chromatography (HPLC). The Organic GC Group Leader is responsible for conducting analyses, troubleshooting instrumentation, performing the required quality control activities, and scheduling analyses so that projects will be completed on-time. The implementation of new methods will be required as new environmental regulations mandate or allow their use.

Background/Educational Requirements

The Organic GC Group Leader will possess a science degree, preferably in chemistry, and/or suitable work experience. The work experience will include several years of performing organic analyses and a demonstrated ability to supervise and train organic staff members.

Duties and Responsibilities

1. Responsible for the productivity and quality of organic chemistry projects and data.
2. Operation, maintenance, and troubleshooting of instrumentation.
3. Quality control program implementation and maintenance.
4. Reviewing organic data.
5. Developing schedules for in-house work that will allow on-time report generation.
6. Maintenance of acceptable productivity.
7. Supervision and technical development of employees.
8. Improving all aspects of operations while emphasizing quality and efficiency.
9. Developing and researching new and better procedures.
10. Performing analyst and method certification as needed.
11. Maintaining a complete lab notebook in an organized manner.
12. Adhering to lab safety procedures.
13. Keeping lab neat and clean.
14. Perform all other duties as deemed necessary by management.

3.5.2.3 Inorganic Group Leader

Job Description

The Inorganic Group Leader performs a variety of analyses in the metals and/or non-metals laboratory. The Inorganic Group Leader is responsible for conducting analyses, troubleshooting instrumentation, performing the required quality control activities, and scheduling analyses so that projects will be completed on-time. The implementation of new methods will be required as new environmental regulations mandate or allow their use.

Background/Educational Requirements

The Inorganic Group Leader will possess a science degree, preferably in chemistry, and/or suitable work experience. The work experience will include several years of performing inorganic analyses and a demonstrated ability to supervise and train inorganic staff members.

Duties and Responsibilities

1. Responsible for the productivity and quality of inorganic chemistry projects and data.
2. Operation, maintenance, and troubleshooting of instrumentation.
3. Quality control program implementation and maintenance.
4. Reviewing inorganic data.
5. Developing schedules for in-house work that will allow on-time report generation.
6. Maintenance of acceptable productivity.
7. Supervision and technical development of employees.
8. Improving all aspects of operations while emphasizing quality and efficiency.
9. Developing and researching new and better procedures.
10. Performing analyst and method certification as needed.
11. Maintaining a complete lab notebook in an organized manner.
12. Adhering to lab safety procedures.
13. Keeping lab neat and clean.

14. Perform all other duties as deemed necessary by management.

3.5.2.4 Client Services Group Leader

Job Description

The Client Services Group Leader function is to manage the members of the Project Chemist group as well as fulfilling the duties of a Project Chemist. The Client Services Group Leader will provide training and support to the client services staff.

The emphasis of the client service role is on being the technical liaison between the client and the laboratory. The client service representative is responsible for providing our clients with prompt, courteous, and responsive service.

The Client Services Group Leader must be a good organizer and capable of doing some passive marketing. This position will be within the Client Services area and will report to the Client Services Manager.

Background/Educational Requirements

The Client Services Group Leader will possess a B.A. or B.S. degree in chemistry or related laboratory science. The work experience will include at least two years as a client service representative, demonstrated leadership skills, and knowledge of the methods used in the environmental testing industry.

Duties and Responsibilities

1. Maintenance of acceptable productivity.
2. Maintenance of a high-quality work product.
3. Consulting with clients and meeting the clients' needs.
4. Working with appropriate Lab Managers to schedule work.
5. Supervising the members of the client services group.
6. Monitoring and making sure client due dates are met.

7. Developing staff members through training and coaching.
8. Marketing new clients via phone calls and follow-up literature.
9. Holding regularly-scheduled meetings to keep staff members informed.
10. Perform all other duties as deemed necessary by management.

3.5.2.5 Senior Inorganic Chemist

Job Description

The Senior Inorganic Chemist performs inorganic chemical analyses accurately and efficiently in the metals and/or non-metals laboratories. This chemist follows established laboratory methods and performs all Quality Control practices applicable to those methods. The Senior Inorganic Chemist also assists in method development, instrument troubleshooting, and the training of analysts or chemists as needed.

Background/Educational Requirements

The Senior Inorganic Chemist will possess a degree in Chemistry and several years of suitable work experience. The Senior Inorganic Chemist will have proven experience in developing and implementing new analytical techniques.

Duties and Responsibilities

1. Obtaining the knowledge and ability to perform all necessary inorganic analyses.
2. Acquiring the ability to calibrate, operate, and maintain all instruments/apparatus required to perform the inorganic analyses.
3. Performing routine care and maintenance of the instruments/apparatus utilized in conducting the inorganic procedures.
4. Performing all QA/QC procedures that have been outlined for a particular test.
5. Maintaining a high level of productivity and quality.

6. Certifying all new analytical methods via the method certification guidelines as outlined in the Quality Control Manual.
7. Maintaining an individual lab notebook.
8. Following all Laboratory Safety Procedures.
9. Maintaining a clean and organized work area.
10. Perform all other duties as deemed necessary by management.

3.5.2.6 Inorganic Chemist

Job Description

Performs classical chemical analyses accurately and efficiently, following established laboratory methods, while following all Quality Control Practices applicable to those methods.

Background/Educational Requirements

Requires a degree in Chemistry and/or related field with at least two years of College Chemistry and work experience.

Duties and Responsibilities

1. Performing all inorganic chemical analyses in an accurate and efficient manner.
2. Maintaining a high level of productivity and quality.
3. Performing analyst and method certifications as needed.
4. Carrying out all required QC procedures and make sure results fall within accepted limits or are qualified appropriately.
5. Keeping all worksheets and calculations up-to-date.
6. Maintaining a complete lab notebook in an organized manner.
7. Troubleshoot problems with instrumentation/equipment.
8. Performing routine maintenance and care of laboratory equipment.
9. Keeping track of and meeting all hold times and due dates.
10. Following lab safety procedures.
11. Keeping lab neat and clean.
12. Developing and researching new and better procedures.

13. Assisting in the scheduling of projects as they come in.
14. Improving all aspects of the job, while emphasizing quality and efficiency.
15. Perform all other duties as deemed necessary by management.

3.5.2.7 Organic Chemist

Job Description

The Organic Chemist performs organic analyses on environmental samples utilizing gas chromatography, gas chromatography mass spectrometry, and/or HPLC. The Organic Chemist is responsible for conducting analyses, troubleshooting instrumentation and performing the required quality control activities. The implementation of new methods will be required as new environmental regulations mandate their use.

Background/Educational Requirements

The Organic Chemist will possess a B.S. in chemistry or a related field. Work experience may contain analytical experience with environmental samples.

Duties and Responsibilities

1. Performing all organic chemical analyses in an accurate and efficient manner.
2. Maintaining a high level of productivity and quality.
3. Performing analyst and method certifications as needed.
4. Carrying out all required QC procedures and make sure results fall within accepted limits or are qualified appropriately.
5. Keeping all worksheets and calculations up-to-date.
6. Maintaining a complete lab notebook in an organized manner.
7. Troubleshoot problems with instrumentation/equipment.
8. Performing routine maintenance and care of laboratory equipment.
9. Keeping track of and meeting all hold times and due dates.

10. Following lab safety procedures.
11. Keeping lab neat and clean.
12. Developing and researching new and better procedures.
13. Assisting in the scheduling of projects as they come in.
14. Improving all aspects of the job, while emphasizing quality and efficiency.
15. Perform all other duties as deemed necessary by management.

3.5.2.8 Organic Extraction Chemist

Job Description

The Organic Extraction Chemist will perform environmental sample extractions in preparation for the analysis of these samples for organic compounds. The Organic Extraction Chemist will be thoroughly familiar with all EPA SW-846 methods utilized by TriMatrix for the extraction of environmental samples. The Organic Extraction Chemist is responsible for conducting extractions, troubleshooting extraction problems and performing all quality control activities required by the methodologies. The Organic Extraction Chemist will establish a schedule for extractions that will allow all samples to be extracted within EPA hold times.

Background/Educational Requirements

The Organic Extraction Chemist will possess a four year college degree in chemistry or a related field. Work experience may include performing sample extractions on environmental samples.

Duties and Responsibilities

1. Performing all organic extractions in an accurate and efficient manner.
2. Maintaining a high level of productivity and quality.
3. Performing analyst and method certifications as needed.
4. Carrying out all required QC procedures.
5. Keeping all worksheets and calculations up-to-date.

6. Maintaining a complete lab notebook in an organized manner.
7. Troubleshoot problems with instrumentation/equipment.
8. Performing routine maintenance and care of laboratory equipment.
9. Keeping track of and meeting all hold times and due dates.
10. Following lab safety procedures.
11. Keeping lab neat and clean.
12. Developing and researching new and better procedures.
13. Assisting in the scheduling of projects as they come in.
14. Improving all aspects of the job, while emphasizing quality and efficiency.
15. Perform all other duties as deemed necessary by management.

3.5.2.9 Sample Coordinator

Job Description

The Sample Coordinator is a support member to the Client Services staff. The Client Services area is responsible for providing highly-responsive and quality service to our clients. The Client Services area is the interface between our clients and our lab. The Sample Coordinator is responsible for logging in all samples received in the laboratory into our LIMS system. This function entails receiving, sorting, identifying, and numbering samples received by the lab.

Background/Educational Background

Associates degree and/or Bachelors degree in the sciences or business. This person needs to be highly organized, innovative and motivated. A working knowledge of the EPA methods used in the laboratory is helpful.

Duties and Responsibilities

1. Logging in all samples in an accurate and efficient manner.
2. Maintaining a high level of productivity and quality.
3. Checking pH of all applicable incoming samples.

4. Monitoring the integrity of all samples received by the lab (hold times, broken samples, etc.).
5. Maintaining complete chain-of-custody of samples.
6. Numbering all incoming samples.
7. Providing all internal clients with logs of samples received in the lab.
8. Initiating some projects on LIMS.
9. Making up bottle orders for new projects.
10. Following lab safety procedures.
11. Keeping lab neat and clean.
12. Developing and researching new and better procedures.
13. Assisting in the scheduling of projects as they come in.
14. Improving all aspects of the job, while emphasizing quality and efficiency.
15. Perform all other duties as deemed necessary by management.

3.5.2.10 Project Technician Bottle/Cooler Preparation

Job Description

The role of the Project Technician is to be a support member to the Client Services staff. The Client Services area is responsible for providing highly responsive and quality service to our clients. The Client Services area is the interface between our clients and our lab. This Project Technician is responsible for correctly preparing and shipping to the client, sample coolers containing the sampling containers requested. This person must maintain an inventory of bottles as well as monitor the integrity of all the sample coolers. This position reports to the Group Leader - Project Technician and the Client Services Manager.

Background/Educational Background

High school diploma and an Associates degree in the sciences is preferable. This person needs to be highly organized, innovative and motivated.

Duties and Responsibilities

1. Maintaining acceptable productivity.
2. Maintaining high quality work product.
3. Ordering and maintaining an ample supply of sample containers and accessories.
4. Maintaining cooler inventory.
5. Maintaining cleanliness of coolers.
6. Keeping work area in a clean, functional environment.
7. Providing price comparisons on products ordered, to provide us with a competitive pricing on supplies ordered.
8. Providing support to the Project Chemists and sample login when needed.
9. Perform all other duties as deemed necessary by management.

3.5.2.11 Data Entry Clerk

Job Description

The main function of the Data Entry Clerk is to input data received from the laboratory area into the LIMS system. This will include all analytical data, quality control data and any necessary comments. The Data Entry Clerk will be responsible for entering high quality (error free) data.

Background/Educational Requirements

High school diploma, good English and data entry skills. This person should be organized, motivated and have data entry background.

Duties and Responsibilities

1. Maintaining acceptable productivity.
2. Maintaining high quality work product.
3. Entering all benchsheet information (this includes entering and verifying data).
4. Continued training on LIMS.

5. Miscellaneous jobs such as filing reports, etc.
6. Perform all other duties as deemed necessary by management.

3.5.2.12 Project Chemist

Job Description

The primary duty of the Project Chemist is to be the technical liaison between the client and the laboratory. The Project Chemist is responsible to provide our clients with prompt, courteous, and responsive service. The Project Chemist will be responsible for all of the client's technical and administrative needs and therefore will be evaluated on how he/she has satisfied those identified needs. This position is part of the Client Services group and reports to the Group Leader of Project Chemists and Client Services Manager.

Background/Educational Requirements

B.A. or B.S. degree in chemistry or related laboratory science and 1-2 years environmental lab testing or environmental related experience. A working knowledge of the methods used in the environmental testing field is required as well as good interpersonal skills. Must be able to communicate clearly and accurately.

Duties and Responsibilities

1. Maintaining acceptable productivity.
2. Maintaining high quality work product.
3. Consulting with clients on their needs and our abilities.
4. Working with appropriate laboratory managers to schedule work.
5. Marketing of new clients via phone calls and follow-up literature.
6. Perform all other duties as deemed necessary by management.

3.5.2.13 Administrative Assistant

Job Description

The Administrative Assistant's role is to provide support services management, the client service area, and laboratory personnel, fulfilling duties such as word processing, work received log, proposal log, billings log, accounts receivable mailing, etc. The Administrative Assistant is a part of the client services and reports to the Client Services Supervisor.

Background/Educational Background

Associates Degree/and or Bachelors Degree in a business related field and 2-3 years word processing experience. This person needs to be highly organized, motivated and innovative. The person should have a good grasp on the English language and excellent word processing skills.

Duties and Responsibilities

1. Maintaining acceptable productivity.
2. Maintaining high quality work product.
3. Word processing.
4. Maintaining work received log.
5. Maintaining proposal log.
6. Maintaining monthly billing logs.
7. Maintaining accounts receivable log.
8. Answering phones.
9. Compiling marketing information.
10. Perform all other duties as deemed necessary by management.

3.5.2.14 Laboratory Field Technician

Job Description

The Laboratory Field Technician is a support member to the client services staff. The Laboratory Field Technician is responsible for taking water samples at homes, making deliveries, and doing sample pick ups for the lab. This position reports to the Client Services Manager.

Background/Educational Background

A drivers license and a good driving record. This person needs to be flexible with hours and be able to make trips on short notice.

Duties and Responsibilities

1. Taking samples in homes as a part of our water test program.
2. Making deliveries of coolers, bottles, quotes, etc. for the laboratory.
3. Picking up samples on a regular basis at our Wixom office and other miscellaneous locations.
4. Perform all other duties as deemed necessary by management.

3.5.2.15 Laboratory Computer Systems Manager

Job Description

Provide technical review, guidance and training in all current and future laboratory computer applications.

Background/Education Requirements

Requires a degree in computer sciences with an emphasis in a chemistry or general science curriculum.

Duties and Responsibilities

1. Developing a complete understanding of the Laboratory Information Management System (LIMS).
2. Reviewing all laboratory computer applications and processes, including all instrument computer interfaces, data transmission/archiving processes and document control.
3. Providing database maintenance support activities for the LIMS system.
4. Providing technical direction and orchestrating implementation of a electronic storage systems for the laboratory.

5. Developing and implementing LIMS instrument interface activities for each laboratory area.
6. Providing technical training of the laboratory staff in software applications and basic computer operational activities.
7. Perform all other duties as deemed necessary by management.

3.5.2.16 Laboratory Computer Technician

Job Description

Provide technical assistance and support in all current and future laboratory computer applications.

Background/Educational Requirements

Requires experience in computer application development and/or satisfactory progress towards a computer science degree.

Duties and Responsibilities

1. Developing a complete understanding of the Laboratory Information Management System (LIMS).
2. Providing database maintenance support activities for LIMS.
3. Providing end user hardware and software support.
4. Performing maintenance programming within the LIMS system.
5. Perform all other duties as deemed necessary by management.

3.5.2.17 LIMS Data Coordinator

Job Description

The LIMS Data Coordinator has the responsibility of providing support and maintenance activities for the Laboratory Information Management System (LIMS). LIMS support activities will involve assisting the Laboratory Managers and staff members in whatever way possible. LIMS maintenance activity includes upkeep of the method, parameter and test tables as well as coordination of optical disk archiving activities.

Background/Education Requirements

The LIMS Data Coordinator will possess at a minimum a high school diploma and two years of college in a related curriculum. Work experience will include a minimum of 3 years in the environmental laboratory business and some working knowledge of computer systems and software.

Duties and Responsibilities

1. Providing maintenance activities for the LIMS method, parameter and test tables.
2. Providing administrative assistance to the Laboratory Manager for LIMS related activities.
3. Coordinating archiving activities involved with the laboratory electronic storage systems.
4. Perform all other duties as deemed necessary by management.

3.6 RESUMES-KEY STAFF MEMBERS

Laboratory Manager
Quality Assurance Manager
Project Chemist Manager
Organic Laboratory Manager
Inorganic Laboratory Manager

DOUGLAS E. KRISCUNAS — Laboratory Manager

EDUCATION

B.S., Environmental Sciences, Grand Valley State University, 1976

PROFESSIONAL SUMMARY

Mr. Kriscunas is responsible for the accuracy and integrity of all analytical data finalized at this location. He is continuously available for client support to resolve analytical issues as they pertain to environmental problems.

PROFESSIONAL EXPERIENCE

- **Detroit, Michigan.** Laboratory Supervisor for a field laboratory established at the Detroit Wastewater Treatment Plant. The project involved a one-year pilot study of the overall operation and plant performance to upgrade and modify existing treatment processes to meet current and future discharge limits. Approximately 20,000 samples were analyzed by seven full-time analysts.

- **Edmore, Michigan. Hitachi Magnetics Corporation.** Participated in the development and implementation of an on-site, flow-through bioassay of the plant discharge. The study was performed in conjunction with the Michigan Department of Natural Resources, Water Quality Division.

- **Grand Rapids, Michigan. EDI Laboratory Certification.** Direct responsibility for the inorganic parameters analysis and quality control measures necessary for laboratory certification under the Safe Drinking Water Act (SDWA) of 1974. Certification involved both analysis of unknown control samples and corresponding on-site evaluation by the U.S. EPA Region V laboratory certification team.

- **Muskegon, Michigan. Uniroyal Chemical Company.** Participated in the soil survey and on-site evaluation of potential soil contamination from deposited chemical waste materials produced by a major chemical company. On-site sample analyses for select parameters were made to locate and detail the extent of contamination.

- **Edmore, Michigan. Hitachi Magnetics Corporation.** Participated in the implementation of a treatability study to effectively remove cobalt and samarium from industrial waste. The study results led to the design and installation of treatment facilities.

- **Columbia, Missouri. A.B. Chance Corporation.** Responsible for implementing a treatment study for effective removal of heavy metals from process wastewater in order to achieve acceptable discharge limits.

- **Kent County, Michigan. Mill Creek Watershed Management Project.** Participated in the collection, mapping and interpretation of environmental characteristics to be used as prototype guidelines for the management of areawide streams in the Great Lakes Basin. The project was funded by the Environmental Protection Agency.

- **Three Rivers, Michigan. Hydramatic Division, General Motors Corporation.** Responsible for the analytical services conducted on a survey of process wastewater for an automotive transmission manufacturer. The project involved data collection and analytical services including grab samples, setting automatic samplers on an hourly basis for a seven-day period, and installing recording meters for continuous pH monitoring.

- **Grand Rapids, Michigan. Michigan Department of Public Health Laboratory Certification.** Supervised analytical, bacteriological and quality control activities involved in achieving certification status for the analysis of potable water supplies in Michigan.

- **Higgins Lake, Michigan. Ralph MacMullan Conference Center.** Served on a three-member panel before a meeting of the Northern Michigan Environmental Health Association. The topic of discussion was an overview of organic chemicals now found in much of Michigan's ground waters. A representative from industry and the MDPH laboratory completed the panel.

- **Grand Rapids, Michigan. Haviland Chemical Company.** Coordinated a static bioassay performed on a water-based detergent utilizing fathead minnows in the 96-hour static test.

- **Sparta, Michigan.** Conducted a dendrological survey of a proposed oil drilling site. The survey was incorporated in an overall environmental assessment of the proposed drilling site.

- **Caledonia, Michigan.** Conducted a dendrological survey of riparian vegetation types located along the banks of the Thornapple River in the area of the Labarge Dam.

- **Grand Haven, Michigan.** Conducted a limnological investigation of the estuary waters of the Grand River watershed near Grand Haven. The collected limnological data were evaluated for potential eutrophication problems resulting from nutrient discharges upstream.

- **Kalamazoo, Michigan. American Cyanamid Company.** Supervised laboratory work required in assisting a major chemical manufacturer with a permit application for existing facility hazardous waste management operation to administratively complete four supplemental technical attachments, multidisciplinary services were required in the areas of hydrogeologic investigation, environmental assessment, failure mode assessment, and engineering review. Field work was completed in 19 days with a report to the client in 25 days to meet scheduled deadlines.

- **Kent County, Michigan.** Coordination of field and laboratory services in conjunction with Act 641 monitoring requirements at two county-owned and operated refuse sites.

Specialized studies were also conducted to identify possible use of landfill gases for electric power generation and the source identification of volatile organic contaminants typical of most municipal landfills.

- **Cascade Township, Michigan. Cascade Resource Recovery/Waste Management, Inc.** Implementation of two separate tracer studies aimed at pinpointing possible cracks or defects in the clay liners of four hazardous waste disposal trenches. The study utilized a low absorptivity fluoroscene water soluble dye introduced to each trench. Samples collected from each liner failure detection system was then analyzed for the fluorescent characteristics of the dye.

- **Cascade Township, Michigan. Cascade Resource Recovery/Waste Management, Inc.** Coordination of field and laboratory services in connection with Michigan Department of Natural Resources Act 64 and U.S. EPA RCRA monitoring requirements. Each sampling event involves collection of ground waters, surface waters, and leak detection monitoring sites.

- **Cascade Township, Michigan. Cascade Resource Recovery/Chemical Waste Management, Inc.** Acted as project chemist and field services coordinator for activities involved in the excavation and site decontamination of an Act 64/RCRA hazardous waste disposal facility. The decontamination program involved the analysis of soils collected in and around each disposal trench after the removal of approximately 20,000 cubic yards of waste materials.

- **Cincinnati, Ohio. Rumpke Waste Systems, Inc.** Acting project manager for a large waste disposal firm headquartered in Ohio, with 20+ landfills located in a 5 state geographical area. Mr. Kriscunas is responsible for coordination of laboratory activities in conjunction with all ground water, surface water and NPDES monitoring requirements.

RICK D. WILBURN — Quality Assurance Manager

EDUCATION

B.S., Environmental Studies, Earlham College, 1985

PROFESSIONAL SUMMARY

Mr. Wilburn is responsible for all aspects of the laboratory Quality Control/Quality Assurance Program. Primary responsibilities include conducting internal and external auditing of the laboratory, procurement and maintenance of state and federal certifications, and ensuring that all facets of the quality control program remain at the highest level possible. Mr. Wilburn also manages the external and internal Quality Control check sample programs.

PROFESSIONAL EXPERIENCE

- **TRACE Analytical Laboratories, Inc. - Quality Assurance Manager from 12/95 to 10/96.** Responsible for designing, implementing, and monitoring a formal quality control program. The program included: conducting internal and hosting external audits, implementing corrective actions resulting from any deficiencies, scheduling and reporting performance evaluation sample results, and the review of all Level 5 data packages.

- **EARTH TECH - Organic Laboratory Manager, from 10/95 to 12/95.** As Organic Laboratory Manager, Mr. Wilburn was responsible for the day to day operations of the organic laboratory, including volatile and semi-volatile analyses by gas chromatography and gas chromatography/mass spectrometry. His responsibilities included scheduling, instrument maintenance, the writing and implementation of standard operating procedures, quality assurance, analytical data review, the technical development of all the organic laboratory personnel, and project management. Mr. Wilburn was also responsible for research and development in the organic laboratory, focusing on ways to automate and improve sample analysis, data quality, and turnaround time.

- **EARTH TECH (Formerly WW Engineering & Science) - Semi-Volatile Laboratory Supervisor, from 1/94 to 10/95.** Responsible for the daily operation of the semi-volatile laboratory. The semi-volatile laboratory utilizes gas chromatography, gas chromatography/mass spectrometry and high performance liquid chromatography in the analysis of semi-volatile organic compounds.

- **WW Engineering & Science - Supervisor, Organic Extraction Laboratory, from 4/93 to 1/94.** Supervisor of the staff of chemists responsible for all organic extractions. Accountable for the processing, quality, and turn around of a wide variety of samples involving many extraction techniques and methodologies. Continually experimenting with

automation and new technologies to improve extraction quality and turn around time, including solid phase and supercritical fluid extractions.

- **WW Engineering & Science - Supervisor, Mass Spectrometry Laboratory, from 9/89 to 1/94.** Supervisor of the staff of chemists analyzing samples for semi-volatile organics in the mass spectrometry laboratory. Oversee all analysis and daily activities involved with the mass spectrometry laboratory. Evaluate, recommend and implement new technologies. Implementations of these include sub-ambient injections using a Varian SPI injector, sub-ambient temperature programs for optimized chromatography, and the use of ion trap mass spectrometers for lower operating detection limits

- **IT Corporation, (formerly PEI Associates, Inc.) - Chemist, Level 3, GC/MS Semi-Volatile Team Leader, from 7/88 to 9/89.** Along with daily analysis of samples, responsible for coordinating the efforts of the three analysts and three instruments used for semi-volatile analysis. This included scheduling each instrument/analyst to make sure analyses were completed correctly and on time, training new personnel, instrument maintenance, data checking, and reporting project results to management for client distribution. Leader of GC/MS Quality Circle group.

- **PEI Associates, Inc. - Chemist, Level 2, GC/MS Analyst, from 12/86 to 7/88.** Primary responsibilities included analyzing soil, water, and other media with an Extrel ELQ-400 mass spectrometer system. Analyses performed included semi-volatile and volatile organics listed on the EPA's Toxic Compounds List according to the Contract Laboratory Program protocol. Also analyzed various other non - Toxic Compounds List compounds using appropriate methods.

- **PEI Associates, Inc. - Chemist, Level 1, GC Analyst, from 7/85 to 12/86.** Carried out a variety of organic analyses in a wide range of matrixes. Was a primary analyst conducting CLP testing for pesticides and PCB's, and was the primary analyst for routine and non-routine testing for herbicides, and volatile organics.

GARY L. WOOD — Client Service Manager

EDUCATION

B.S. Business Administration, Aquinas College

PROFESSIONAL SUMMARY

As Client Services Manager from 1/96 to present, Mr. Wood is responsible for the overall management of the laboratory office, data entry, login, bottle prep and project chemist team. His responsibilities as a manager includes scheduling, quality assurance, report generation and review, and the technical development of all client services personnel. Mr. Wood is also responsible for the overall management of a variety of laboratory clients. Mr. Wood oversees all aspects of projects, including proposals, sample flow, analytical data, quality assurance and billing. He also serves as a client consultant on matters pertaining to laboratory issues such as test/method selections as they relate to current state and federal regulations. Mr. Wood's project experience, with a clientele ranging from small engineering firms to large industrial corporations, has enabled him to acquire a strong and diverse working knowledge of client needs in the laboratory.

- **Group Leader-Client Services Group from 8/94 to 1/96.** In addition to all responsibilities of a project chemist, was also responsible for the direct coordination, management, technical development of the project management group.

- **Project Chemist from 9/92 to 8/94.** Responsible for complete management of a variety of laboratory projects and clients. Primary responsibilities included client development and consulting, project proposals, project initiation, and tracking, report generation and review, and project follow-up with clients.

- **Organic Analyst from 7/89 to 9/92.** Responsible for the analysis of volatile and semi-volatile organics by GC and HPLC utilizing EPA approved methods. Responsibilities included instrument maintenance and troubleshooting as well as the training of new staff members. Was involved in bringing the Turbochrome Computer system online.

- **Inorganic Analyst from 7/87 to 7/89.** Performed laboratory analyses using titration, distillation, gravimetric, and various other methods. Instruments included for pH meters, conductivity meters, dissolved oxygen meters, TOC analyzer, TOX analyzer, and UV/VIS Spectrophotometer.

- **Technician (part-time) from 10/84 to 1/86.** Worked in login and receiving, bottle preparation, and special projects. Performed field services such as soil and water sampling and was also involved in special projects for field services.

JEFFREY P. GLASER — Organic Laboratory Manager

EDUCATION

B.S., Biochemistry, Michigan State University, 1987

PROFESSIONAL SUMMARY

Mr. Glaser has 7 years of environmental laboratory experience, including over 4 years of supervisory experience. He is currently responsible for the operation and management of the Organic Laboratory, including sample preparation, semi-volatiles, and volatiles. Main functions include supervision and training of personnel, formulation of standard operating procedures, final approval of all organic laboratory data, and laboratory purchase approval.

Mr. Glaser served one and a half years as the Laboratory Manager of the Muskegon facility. He was responsible for all aspects of laboratory performance. This included, but was not limited to, analytical testing and reporting; business development; customer service; capital expenditures, quality control; quality assurance; laboratory safety; and laboratory profitability. He was responsible for the hiring, training, guidance, and evaluation of all laboratory personnel, and for direction of overall laboratory policies and practices.

PROFESSIONAL EXPERIENCE

- **Great Lakes Environmental Laboratories - Laboratory Manager.** Responsible for all aspects of laboratory performance. This included, but was not limited to, analytical testing and reporting; business development; customer service; capital expenditures, quality control; quality assurance; laboratory safety; and laboratory profitability. He was responsible for the hiring, training, guidance, and evaluation of all laboratory personnel, and for direction of overall laboratory policies and practices.

- **Great Lakes Environmental Laboratories - Senior Chemist.** Mr. Glaser's responsibilities included supervision and training of other laboratory personnel, coordination of sample workloads, data review and evaluation, and quality control. He was also responsible for analysis of pesticides, PCB's, and herbicides using an HP 5890 GC w/ECD detectors.

- **Anatech Analytical Laboratories - Semi-Volatile GC/MS Operator.** Mr. Glaser operated and maintained a Hewlett Packard GC/MSD UNIX-based Chem Station. He was responsible for all semi-volatile GC/MS analysis and new method development. He served as the Organic Supervisor for the first quarter of 1991.

- **Anatech Analytical Laboratories - Volatile GC/MS Operator.** Mr. Glaser operated and maintained a Finnigan Ion Trap GC/MS system consisting of a Varian GC and a Tekmar purge and trap autosampler. Primary methodology used was 624/8240.

- **Anatech Analytical Laboratories - Volatile Organic Chemist.** Mr. Glaser was responsible for operation and maintenance of two volatile GC systems utilizing ELCD, FID, and PID detectors, and Tekmar and O.I. Analytical purge and trap autosamplers. Primary analyses were 601 and 602.

JT WHITMORE — Inorganic Laboratory Manager

EDUCATION

B.S. Health Science - Medical Technology, Grand Valley State University, 1987
[emphasis in analytical and clinical laboratory studies]
2nd Major Technical Chemistry, Grand Valley State University
[emphasis in instrumental analysis]

PROFESSIONAL SUMMARY

As Inorganic Laboratory manager, Mr. Whitmore is responsible for the daily operation of the metals and inorganic laboratory areas. His primary responsibilities include operational, personnel, and organizational management. Other duties include creation and maintenance of standard operating procedures for both areas, method development, trouble shooting current and new equipment, and working with the Client Services group to ensure the production of high-quality data in the most efficient manner.

PROFESSIONAL EXPERIENCE

- **EARTH TECH - Metals Chemist from 5/90 to 11/96, Later as TriMatrix Laboratories, Inc. from 11/96 to 2/97.** Responsible for training all new chemists and analysts, preparation of working standards, data backup of computers, computer repair, instrument maintenance and repair, method development, quality control cheeks on data, and preparation of samples for analysis in the metals lab using EPA methods for the graphite furnace, flame atomic absorption, mercury cold vapor, and inductively-coupled plasma.

- **Continental Bio-Laboratories - Lab Technician from 5/84 to 5/90.** Clinical analysis of biological diseases by a variety of methods including:

Microbiology	-setup, culture, and identification of organism by plate technique and by automated ID unit.
Hematology	-blood component analysis
Urinalysis	-microbial and morphologic analysis
Virology	-viral tissue culture
Special Chemistry	-blood gases, electrolytes, drug screen

3.7 APPROVED SIGNATORIES

Designated laboratory staff members have been assigned the responsibility of validating laboratory documents on behalf of the laboratory organization. The general categories or documents that require a valid signature are presented below.

3.7.1 Client/Invoice Reports

All laboratory reports compiled and mailed contain at least one representative signature validating the contents of the laboratory report. Approved signatures for the client/invoice report include the Laboratory Manager, Client Services Manager, Technical Director, Quality Assurance Manager, and the appropriate project chemist. Only these individuals are approved to perform this signatory approval.

3.7.2 Proposals, Price Quotations, and Laboratory Contracts

Proposals or price quotations for laboratory services contain at least one representative signature which validates the pricing, terms and conditions of the quotation. At least one representative signature is required. Approved signatures for proposals and price quotations included the Laboratory Manager, Client Services Manager, project chemists and a sales or marketing representative.

In conjunction with many proposals and quotations, the laboratory may decide to include the laboratory Professional Services Agreement (PSA) in conjunction with the quotation. The PSA can only be signed by the Laboratory Manager.

Required signatures for laboratory contracts include the Laboratory Manager and a marketing representative.

3.7.3 Quality Assurance Project Plans (QAPP)

Quality Assurance Project Plans contain representative signatures of several responsible parties outside the laboratory. The only laboratory signature generally

found on a QAPP is that of the quality assurance supervisor. The QA Manager has designated QA/QC responsibilities that are fully documented in the QAPP documents. All QAPP are signed prior to submission to a governing body or client.

The signatures on the QAPP ensure that all procedures, materials, quality control practices and project reports meet the pre-defined goals of the plan.

3.7.4 Purchase Orders and Agreements

Because the laboratory spends a significant portion of it's annual budget on supplies and equipment, guidelines have been established to document and control purchasing.

Purchasing of general supplies are handled through a contracted vendor within the budgetary guidelines established for each laboratory area.

For major purchases, such as equipment, service assessments, or building renovations in excess of $500.00, purchase orders or agreements must be approved by the Laboratory Manager or CEO.

3.7.5 Binding Statements - Laboratory Certification Documents or Accreditation

Certain certification or accreditation programs require the laboratory to provide items and statements regarding many details on the laboratories operations and staff.

In some cases these statements must be presented to the certifying body accompanied by a binding signature of a corporate executive within TriMatrix .

3.8 ACCREDITATION'S, CERTIFICATION'S, AND PROFICIENCY PROGRAMS

3.8.1 Laboratory Certification - Federal, State and Industrial

TriMatrix maintains Federal, State and Independent certifications and accreditation's and participates in analytical proficiency studies at the federal, state and industrial level. These programs include the following:

3.8.1.1 Federal Programs

USEPA - State of Michigan Water Supply Certification

USEPA - Water Pollution Performance Evaluation Program

USEPA - DMR-QA Performance Evaluation Program

3.8.1.2 State Programs

		Lab I.D.
Michigan	Department of Public Health Chemical Analysis of Potable Water	0034
Minnesota	Department of Health Chemical Analysis of Environmental Samples	026-999-161
North Dakota	Department of Health/Consolidated Laboratories Chemical Analysis of Environmental Samples	R-095
Wisconsin	Department of Natural resources Chemical Analysis of Environmental Samples	000472650
Florida	Department of Environmental Protection Chemical Analysis of Environmental Samples	950066
Pennsylvania	Department of Environmental Protection Chemical Analysis of Environmental Samples	68-534

3.8.1.3 Industrial Programs

Allied Signal
Corporate Environmental Laboratory
Chemical Analysis of Environmental Samples

3M Corporation
Corporate Environmental Laboratory
Chemical Analysis of Environmental Samples

Dow Chemical Corporation

Corporate Environmental Division
Chemical Analysis of Non-Potable Water

E.I. Dupont
Environmental Services Division
Chemical Analysis of Environmental Samples

Ford Motor Corporation
Environmental Quality Office
National Contract Laboratory program

Monsanto Corporation
Chemical Analysis of Environmental Samples

Occidental Chemical Corporation
Chemical Analysis of Environmental Samples

General Electric Corporation
CPA Laboratory Program
Chemical Analysis of Environmental Samples

3.8.2 Method/Analyst Certification

All methods of analysis utilized by TriMatrix are certified or validated prior to their use, whether newly employed or as a replacement for a current methodology. Method certification is analogous with analyst certification, and essentially requires the same steps in order to establish detection limits, reporting limits, and criteria for control limits.

Certifications are performed in three distinct categories:

1). When the analytical method specifies how the analyst and method are certified.
2). In the absence of method certification instructions in the test method.
3). In the absence of analyst certification instructions in the test method.

All certifications are documented, reviewed, and validated in each analyst's employee file in accordance to the procedures outlined in the TriMatrix SOP for analyst training.

Method/analyst certifications for the TriMatrix laboratory are divided into two distinct categories: method specific and non-method specific.

3.8.2.1 Method/Analyst Certification - Method Specified Criteria

Method and analyst certifications for most USEPA organic methods are performed as outlined in each of the analytical methods. All certifications are documented, reviewed, and validated in each analyst's employee file. An example of this certification record is presented in Figure 6.

In conjunction to this procedure, a Method Detection Limit (MDL) study is performed in accordance with 40 CFR; Part 16; Appendix B. Results of each study are reviewed by the appropriate Laboratory Area Manager and the QA department. All MDL studies are performed on reagent water and updated annually or whenever major changes are made in an analytical procedure.

3.8.2.2 Method/Analyst Certification - No Method Specified Criteria

All methods used by TriMatrix which were not developed by TriMatrix and for which there is not an EPA or compendia certification requirement will be certified according to the steps below. Method Certification is contiguous with the certification of the analyst and requires essentially the same analytical program. Method certification is necessary in order to establish detection limits, method application limits, and criteria for control limits. In most cases detection limits and recoveries stated in a method are obtained under ideal conditions and do not reflect real world solutions, i.e. silty well water and industrial effluent versus a drinking water supply. Method certification falls into two categories: 1) Methods being employed for the first time and 2) Methods which are to replace currently certified methods (replacement methods). In either case, analysis of client sample may not proceed until certification has occurred.

A). Linear Range

The first step in certifying a method is to establish the linear range (operating range) of the method. A method may be used only over the range in which it is linear. Some methods do not have linear ranges but curves from which results are calculated. For the moment we will ignore methods with curves. A linear range must be established independent of the method data since instruments can effect the range. Standards and multiple detections will be used for establishing the linear range. For example, a range of 1 to 1000 has 3 decades (3 orders of magnitude). Therefore, a range of 1 to 1000 requires 11 levels of test standards (.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, 1000). Notice that each decade follows the 0.5x to 10x rule, i.e. the area 10 to 100 is covered by 5, 10, 20, 50 and 100. The range to be attempted is dependent on the method, the instrument and the area manager's recommendation. If the responses show linearity, the range has been established. If, however, a curve develops or there appear to be two linear ranges, the standards must be repeated including additional levels to verify the status of the questionable area.

Example Certification Record

TARGET	AMT SPK ug/l	#1 AMT FND ug/l	#2 AMT FND ug/l	#3 AMT FND ug/l	#4 AMT FND ug/l	AVG REC ug/l	TABLE 6 RECOVERY RANGE in ug/l	PASS/ FAIL REC	STD DEV	STD DEV LIMIT	STD DEV PASS/ FAIL	OVERALL PASS/FAIL
Benzene	20.0	19.39	19.76	19.34	19.36	19.46	15.2 -26.0	PASS	0.20	6.9	PASS	PASS
Bromodichloromethane	20.0	20.21	20.25	20.49	20.2	20.29	10.1 -28.0	PASS	0.14	6.4	PASS	PASS
Bromoform	20.0	21.11	20.01	21.17	20.67	20.74	11.4 -31.1	PASS	0.54	5.4	PASS	PASS
Bromomethane	20.0	15.39	16.44	15.04	15.7	15.64	0.0 -41.2	PASS	0.60	17.9	PASS	PASS
Carbon Tetrachloride	20.0	19.11	19.35	18.66	18.22	18.84	17.2 -23.5	PASS	0.50	5.2	PASS	PASS
Chlorobenzene	20.0	19.14	19.49	19.36	19.01	19.25	16.4 -27.4	PASS	0.22	6.3	PASS	PASS
Chloroethane	20.0	18.19	19.01	17.71	16.86	17.94	8.4 -40.4	PASS	0.90	11.4	PASS	PASS
2-Chloroethyl Vinyl Ether	20.0	20.62	20.98	20.76	20.88	20.81	0.0 -50.4	PASS	0.16	25.9	PASS	PASS
Chloroform	20.0	20.54	21.28	20.52	20.01	20.59	13.7 -24.2	PASS	0.52	6.1	PASS	PASS
Chloromethane	20.0	17.43	18.13	16.9	16.41	17.22	0.0 -45.9	PASS	0.74	19.8	PASS	PASS
Dibromochloromethane	20.0	20.24	19.68	20.33	20.27	20.13	13.8 -26.6	PASS	0.30	6.1	PASS	PASS
Dichlorobenzene,1,2-	20.0	19.68	20.1	19.86	19.55	19.80	11.8 -34.7	PASS	0.24	7.1	PASS	PASS
Dichlorobenzene,1,3-	20.0	19.12	19.63	19.55	19.28	19.40	17.0 -28.8	PASS	0.24	5.5	PASS	PASS
Dichlorobenzene,1,4-	20.0	19.12	19.93	19.28	19.05	19.35	11.8 -34.7	PASS	0.40	7.1	PASS	PASS
Dichloroethane,1,1-	20.0	19.86	22.67	18.57	20.2	20.33	14.2 -28.5	PASS	1.71	5.1	PASS	PASS
Dichloroethane,1,2-	20.0	22.31	22.46	22.32	22.49	22.40	14.3 -27.4	PASS	0.09	6.0	PASS	PASS
Dichloroethylene, (trans)-1,2-	20.0	20.18	20.12	18.75	18.69	19.44	13.6 -28.5	PASS	0.83	5.7	PASS	PASS
Dichloroethylene,1,1-	20.0	19.44	20.63	18.57	18.09	19.18	3.7 -42.3	PASS	1.12	9.1	PASS	PASS
Dichloropropane,1,2-	20.0	20.92	20.92	20.94	20.64	20.86	3.8 -36.2	PASS	0.14	13.8	PASS	PASS
Dichloropropylene, (cis)-1,3-	20.0	20.58	20.84	20.98	20.39	20.70	1.0 -39.0	PASS	0.26	15.8	PASS	PASS
Dichloropropylene, (trans)-1,3-	20.0	21.15	21.2	21.76	20.92	21.26	7.6 -32.4	PASS	0.36	10.4	PASS	PASS
Ethylbenzene	20.0	19.4	19.96	19.39	18.82	19.39	17.4 -26.7	PASS	0.47	7.5	PASS	PASS

Figure 6

B). Working Curves

Some methods operate from a curve response, i.e. sodium by emission spectroscopy. The method will indicate the working curve which must be verified. The method with working curves requires a full curve each time an analysis is to be performed.

C). The Generation of the Method Detection Limit

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero and determined from analysis of a sample in a given matrix containing analyte.

Scope And Application

This procedure is designed for applicability in a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete , specific and well defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.

The MDL obtained by this procedure is used to judge the significance of single measurement of a future sample.

The MDL procedure was designed for applicability in a broad variety of physical and chemical methods. To accomplish this, the procedure was made device or instrument independent.

D). Procedure

1. Make an estimate of the detection limit using one of the following:

   - The concentration value that corresponds to an instrument signal/noise ratio in the range of 2.5 to 5. If the criteria for qualitative identification of the analyte is based upon pattern

recognition techniques such as PCB analysis, the least abundant signal necessary to achieve identification must be considered in making the estimate.

- The concentration value that corresponds to three times the standard deviation of replicate instrumental measurements for the analyte in reagent water.

- The concentration value that corresponds to the region of the standard curve where there is a significant change in sensitivity at low analyte concentrations i.e. a break in the slope of the standard curve.

- The concentration value that corresponds to known instrumental limitations.

It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the estimate of the detection limit.

2. Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interfering concentrations are not detected at the method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferent). The interferent concentration is presupposed to be normally distributed in representative samples of a given matrix.

3. - If the MDL is to be determined in reagent water (blank) prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated MDL (Recommend between 1 and 5 times the estimated MDL) proceed to Step 4.

- If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated MDL proceed to Step 4.

  If the measured level of analyte is less than five times the estimated MDL, add a known amount of analyte to bring the concentration of analyte to between one and five times the MDL in the case where an interference is co-analyzed with the analyte.

  If the measured level of analyte is greater than five times the estimated MDL there are two options:

- Obtain another sample of lower level of analyte in same matrix if possible.

- The sample may be used "as is" for determining the MDL if the analyte level does not exceed 10 times the MDL of the analyte in reagent water. The variance of the analytical method changes when the analyte concentration increases above the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.

4. - Take a minimum of seven aliquots of the sample to be used to calculate the MDL and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If blank measurements are required to calculate the measured level of analyte, obtain separate blank measurements for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.

   - It may be economically and technically desirable to evaluate the estimated MDL before proceeding with analysis of seven aliquots as described above. This will: (1) prevent repeating this entire procedure when the costs of analyses are high and (2) insure that the procedure is being conducted at the correct concentration. It is quite possible that an incorrect MDL can be calculated from data obtained at many times the real MDL even though the background concentration of analyte is less

than five times the estimated MDL. To insure that the estimate of the MDL is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower MDL. Take two aliquots of the sample to be used to calculate the MDL and process each through the entire method, including blank measurements as described in section d above. Evaluate these data:

- If these measurements indicate the sample is in the desirable range for determining the MDL, take five additional aliquots and proceed. Use all seven measurements to calculate the MDL.

- If these measurements indicate the sample is not in the correct range, re-estimate the MDL, obtain new sample as in section d above and repeat steps listed.

5. Calculate the variance (S^2) and standard deviation (S) of the replicate measurements, as follows:

$$S^2 = \frac{1}{n-1} [\Sigma(x_1)^2 - (\Sigma x_1)2/n]$$

where the X1; i = 1 to n are the analytical results in the final method reporting units obtained from the n sample aliquots and Σ refers to the sum of the X values from i = 1 to n.

6. - Compute the MDL as follows:

MDL = ^{t}(n-1, 1-d = 0.99) (S)

where:

MDL - the method detection

$^{t}_{(n-1,1-d = .99)}$ = the students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. See Table.

S = standard deviation of the replicate analyses.

- The 95% confidence limits for the MDL derived in step f are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution (X2/df) and calculated as follows:

$$MDL_{lcl} = 0.64\ MDL$$

$$MDL_{ucl} = 2.20\ MDL$$

where MDL_{lcl} and MDL_{ucl} are the lower and upper 95% confidence limits respectively based on seven aliquots.

7. Optional iterative procedure to verify the reasonableness of the estimated MDL and calculated MDL of subsequent MDL determinations.

- If this is the initial attempt to compute MDL based on the estimated MDL in step 3, take the MDL as calculated in step 6, spike in the matrix at the calculated MDL and proceed through the procedure starting with step 4.

- If the current MDL determination is an iteration of the MDL procedure for which the spiking level does not permit qualitative identification, report the MDL as that concentration between the current spike level and the previous spike level which allows qualitative identification.

- If the current MDL determination is an iteration of the MDL procedure and the spiking level allows qualitative identification, use S2 from the current MDL calculation and S2 from the previous MDL calculation to compute the F ratio.

if $S^2_A/S^2_B < 3.05$

then compute the pooled standard deviation by the following equation:

$$\text{S-pooled} = [\frac{6S2A + 6S2B}{12}]\ 1/2$$

if S^2_A/S^2_B >3.05, respike at the last calculated MDL and process the samples through the procedure starting with step 4.

- Use the S-pooled as calculated in step 6 to compute the final MDL according to the following equation:

  MDL = 2.681 (S-pooled)

  where 2.681 is equal to t(12,1-a = .99)

- The 95% confidence limits for MDL derived in step 6 are computed according to the following equations derived from percentiles of the chi squared over degrees of freedom distribution.

  MDL_{lcl} = 0.72 MDL

  MDL_{ucl} = 1.65 MDL

  where $_{lcl}$ and $_{ucl}$ are the lower and upper 95% confidence limits respectively based on 14 aliquots.

E). REPORTING

The analytical method used must be specifically identified by number or title and the MDL for each analyte expressed in the appropriate method reporting units; if the analytical method permits options which affect the method detection limit, these conditions must be specified with the MDL value. The sample matrix used to determine the MDL must also be identified with the MDL value. Report the mean analyte level with the MDL if a laboratory standard or a sample that contained a known amount of analyte was used for this determination. Report the mean recovery, and indicate if the MDL determination was iterated.

If the level of the analyte in the sample matrix exceeds 10 times the MDL of the analyte in reagent water, do not report a value for the MDL.

REFERENCE

40 CFR Part 136 Appendix B, USEPA.

Table of Students' t Values at the 99 Percent Confidence Level

Number of Replicates	Degrees of Freedom (n-1)	Students' t Value
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390
		2.326

F). Laboratory Fortified Blanks (LFB)

Laboratory Fortified Blanks spikes will be carried out over 2 separate days at the specified levels including blanks and calibration standards. The data obtained at the 2x or 5x level (for each certified range) will be used to develop an estimated mean and standard deviation for initial control charts. Once this data has been generated and approved by the QA Manager, the method has preliminary certification and is ready for application to real world samples. These control limits will be updated with every batch of samples until 30 numbers have been developed to establish reliable control limits. After the initial 30 data points, updated control limits will be generated at a specified time period (currently quarterly).

3.8.2.3 Analyst Certification Criteria - Non Method Specific (In the Absence of Analyst Certification Instructions in the Test Method)

Non-Method specific certifications are performed in accordance to the procedures developed by TriMatrix. This process involves the analysis of four standard solutions. Calculate the mean and standard deviation (s) and compare with method specifications. If accuracy and precision are outside method specifications, trouble-shoot the procedure and repeat the analysis of four replicates. When method accuracy and precision information is not available, results of the 4 determinations are evaluated against either laboratory established windows or a 70-130% accuracy and ≤20% RPD precision window. Final review and approval is performed by the Laboratory Area Manager and the QA Manager.

In conjunction to this procedure, a Method Detection Limit (MDL) study is performed in accordance to 40 CFR; Part 16; Appendix B. Results of each study are reviewed by the appropriate lab area supervisor and the QA Supervisor. All MDL studies are performed on reagent-grade water and updated annually.

3.8.3 Training

The proper training of laboratory personnel is an essential part of the overall development of staff capabilities. The documentation of these training procedures will not only provide a record of training activities completed, but will also serve as a guideline for the continual development of staff capabilities. This record will also be useful in the guiding of retraining.

All training documents are contained in a personnel training file, and are under direct management of the Quality Assurance Manager.

The personnel training file is a compendium of documents related to the development of each laboratory employee. Contained within this file are all in-house training documents, copies of academic transcripts or degrees, job descriptions, resumes, external training program certificates, safety training

records and any other materials directly related to an individual employees analytical capabilities.

3.8.3.1 Initiation of Training Documents

All laboratory staff members have a set of training documents provided on the date of hire. These documents are developed and maintained throughout the entire employment period. All documents are initiated by the Quality Assurance Manager. All training documents are presented in the attached Appendix A.

- General information located on Page 1 of the personnel training records are initiated and maintained by the Laboratory QA Manager. This information includes: employee name, initial laboratory position, employee number, date of hire, laboratory area and a detail description of initial primary responsibilities. (See example in Appendix A).

- Position changes are also recorded on page 1 of the personnel training record. Position changes are generally limited to the movement of personnel from one lab to another, or one area of interest to another within the same lab area. (i.e., GC/MS volatiles to GC/MS semi-volatiles).

3.8.3.2 General Training

General training requirements include the following: laboratory introduction and walk-through, company benefits review, safety and chemical hygiene plan, safety exam, safety walk/safety equipment review computer network/eMail and review and initial training of the Laboratory Information Management System (LIMS).

The computer network and LIMS review requires initialization process whereby the new user is added to both systems and assigned

network passwords and codes. The laboratory computing staff perform the network assignments.

3.8.3.3 Training-Quality Assurance

- Quality Assurance training includes all items depicted on Page 3 of the attached example. Each item listed will be discussed in detail.

- Presented to each trainee: Completed benchsheets from their newly assigned lab area, QA memo on QC type descriptions, copy(s) of completed stock standard records, copies of instrument maintenance log sheets, analyst notebook record keeping guidelines and all published Quality Assurance operational memorandums. Area specific forms and procedures will also be discussed and presented at this time. Examples of the presented items can be found in Appendix A.

3.8.3.4 Training-Laboratory Specific

- Laboratory Specific training is generally performed in the employee's newly assigned laboratory area and will cover the following items: introduction to apparatus and equipment, a description and demonstration of paperwork flow, including LIMS paperwork and reporting requirements, a review of the standard formats that are used for the writing of laboratory SOPs scheduled for use by the trainee.

- Training of each method will include: a complete understanding of the SOP, equipment, documentation and QA objectives for a procedure. Once the analyst has a complete understanding of all aspects of the procedure, both the trainee and trainer will document the completion.

3.8.3.5 Training-Analyst Certification

- Analyst/Method certifications will be performed in accordance to method specific or QA manual protocols. The protocol depicted in the QA manual should only be used in the absence of a method specific procedure.

3.8.3.6 Quality Assurance

- Quality Assurance activities involved in the training process are an important part of analyst training. These activities include: proper documentation of all training events as displayed in the attached example, maintenance of the training document to keep it current, and most important, adherence to the Quality Assurance procedures and directives as presented in the method protocols.

3.8.3.7 Responsibilities

- Responsibilities for the training process are defined in the following Table 1.

TABLE 1
PERSONNEL TRAINING RECORD
TRAINING RESPONSIBILITIES

Training Activity/Event	Person/Dept. Responsible
Initiation of Training Records	Quality Assurance Group Rep.
Description of Primary Responsibilities	Lab Area Manager
Laboratory Introduction/Walk-Through	Lab Area Manager/Group Leader
Company Benefits Review	Human Resources
Safety-Chemical Hygiene Plans	Safety Coordinator
Safety Exam (implementation & review)	Safety Coordinator
Safety Walk/Safety Equipment Review	Safety Coordinator
Computer Network/E-Mail	Laboratory Computer Group Rep.
Laboratory Information Management System (LIMS)	Laboratory Computer Group Rep.
QA Manual Review	Quality Assurance Manager
General QA Objectives	Quality Assurance Manager
Benchsheets/Control Windows/Qualifications	Quality Assurance Manager
Chemical Inventory Program	Quality Assurance Manager
Stock Standard Record Procedures	Quality Assurance Manager
Instrument Maintenance Logs	Quality Assurance Manager
Instrument Run Logs	Quality Assurance Manager
Analyst Notebook & Procedures	Quality Assurance Manager
Data Recording & Changes	Quality Assurance Manager
QA Operational Memorandums	Quality Assurance Group Rep.
Data Review & Documentation	Lab Area Manager/Group Leader
Introduction to Apparatus & Equipment	Lab Area Manager/Group Leader
Paperwork Flow-LIMS Training	Lab Area Manager/Group Leader
SOP Review-General Format	Lab Area Manager/Group Leader
SOP Review-Method Specific	Lab Area Manager/Group Leader
Analyst Certifications	Lab Area Manager/Group Leader

3.8.3.8 Continuing Training and Education

TriMatrix is committed to education and training on a continuing basis for employees of the laboratory division. Among the various ways in which continuing training occurs are:

- seminars
- cross-training for additional job responsibilities
- retraining
- method and technology updates

3.8.4 Annual Recertification of Analysts

Recertification for all analysts is performed on an annual basis. This process is performed by means of two steps: The first involves the generation of a LIMS report that displays LCS recoveries for a specified period which compares each recovery to the current LIMS generated laboratory established control windows. This report is reviewed and validated by the appropriate Laboratory Area Manager and the QA Manager. All records are then filed in the employee's personnel file. An example of this document is presented in Figure 7. The second step of the recertification process involves a review of the analytical methods and laboratory SOPs for all test performed by an analyst with their Laboratory Area Manager or designate. Each method review is documented and placed in the employee's personnel file.

05-SEP-97 TRIMATRIX LABORATORIES, INC. 2
ANALYST CERTIFICATION SUMMARY REPORT

Analyst: Gretchen Housekeeper
Parameter: BOD, Carboneceous (5-Day)

Method: Biochemical Oxygen Demand
Reference Citation: USEPA-405.1
Application: WATER
Dates Surveyed: 01-JAN-1997 to 31-MAR-1997

LCS Control Limits: lower: 84 upper: 124

Date of Analysis	Percent Recovery
27-MAR-97	105
26-MAR-97	107
20-MAR-97	102
19-MAR-97	106
13-MAR-97	100
12-MAR-97	113
06-MAR-97	115
05-MAR-97	111
26-FEB-97	108
20-FEB-97	100
19-FEB-97	97
14-FEB-97	104
12-FEB-97	106
06-FEB-97	108
05-FEB-97	104
30-JAN-97	99
29-JAN-97	106
23-JAN-97	97
22-JAN-97	111
16-JAN-97	104
15-JAN-97	104
10-JAN-97	112
09-JAN-97	88
08-JAN-97	90
02-JAN-97	106

Total Number of Data Points = 25 Average Recovery = 104.12

Reviewed By

Area Supervisor: _____ QA/QC Supervisor: _____

Figure 7

3.9 LABORATORY SCOPE OF SERVICES

3.9.1 Method and Matrix Capabilities/Analytical Methodologies

Matrix Capabilities

Common matrices tested include surface water, wastewater, soil, groundwater, solid wastes, and sludges. In addition, analyses have been performed on fish, biota, and air samples on a project basis.

Analytical Methodologies

TriMatrix uses written standard operating procedures (SOPs) which are derived from the current revision of methods specified by the United States Environmental Protection Agency, other federal and state agencies, and professional compendia as listed below:

"Test Methods for Evaluating Solid Wastes" (SW-846) Office of Solid Waste and Emergency Response, U.S. EPA.

Current EPA Contract Laboratory Program (CLP) Protocols for the Analysis of Organic and Inorganic Hazardous Substances.

"Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater," EPA-600/4-82-057.

"Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act," 40 CFR, Part 136.

"Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water," USEPA, EMSL-Cincinnati.

"Annual Book of ASTM Standard," Volumes 11.01 and 11.02, ASTM, Philadelphia, PA.

"Techniques of Water Resources Investigations of the United States Geological Survey, Book 5, Laboratory Analysis," USGS, Washington, D.C.

"Standard Methods for the Examination of Water and Wastewater," APHA, AWWA, WPCF, Washington, DC.

"Official Methods of Analysis," AOAC, Arlington, VA.

"Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020.

HAZWRAP; Hazardous Waste Remedial Action Program; U.S. Department of Energy, Oak Ridge, TN Operations Office.

3.9.2 Detection Limits

The process of quantifying an analyte in an environmental matrix using specific analytical methods must use detection limits as points of reference. The three regions of analyte signal as generated by an instrument are separated by detection limits as described below.

3.9.2.1 Instrument Detection Limit - IDL

The IDL separates the region of no analyte detection from the region of signal detection. The IDL is defined as the smallest signal above background noise that an instrument can detect reliably. The IDL is measured by analyzing replicate standard solutions using reagent grade water as the test matrix. Seven consecutive measurements of standards are performed at 3-5 times the required detection limit concentrations on three non-consecutive days. IDL is determined by multiplying by three the average of the standard deviations of the measured values. The IDL will vary from one instrument to another and should not be used as a reportable detection limit (Figure 8). Determine the IDL once per year for trace metals.

3.9.2.2 Method Detection Limit - MDL

The MDL separates the region of signal detection from the region of qualitative (semi-quantitative) determination (Figure 8). The MDL is defined as the minimum concentration of a substance that can be qualitatively measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL also can be described as the minimum concentration of an analyte that the method can detect in a blank or sample matrix. The MDL is generated by analyzing a low concentration of an analyte, usually at one to five times the expected MDL, in reagent grade water. The analysis is performed seven times, and the MDL is calculated by multiplying the standard deviation times the student "t" value at the 99% confidence level. Determine the MDL once per year for each analyte.

3.9.2.3 Reporting Detection Limit - RDL

The RDL separates the region of qualitative (semi-quantitative) determination from the region of quantitative determination. The RDL is defined as the minimum concentration of an analyte that can be reliably detected within specified limits of precision and accuracy during normal laboratory operating conditions. The RDL for an analyte is the MDL multiplied by a factor which varies, depending on the test matrix. A factor of three to five is frequently used. (Figure 8).

Regions of Analyte Signal

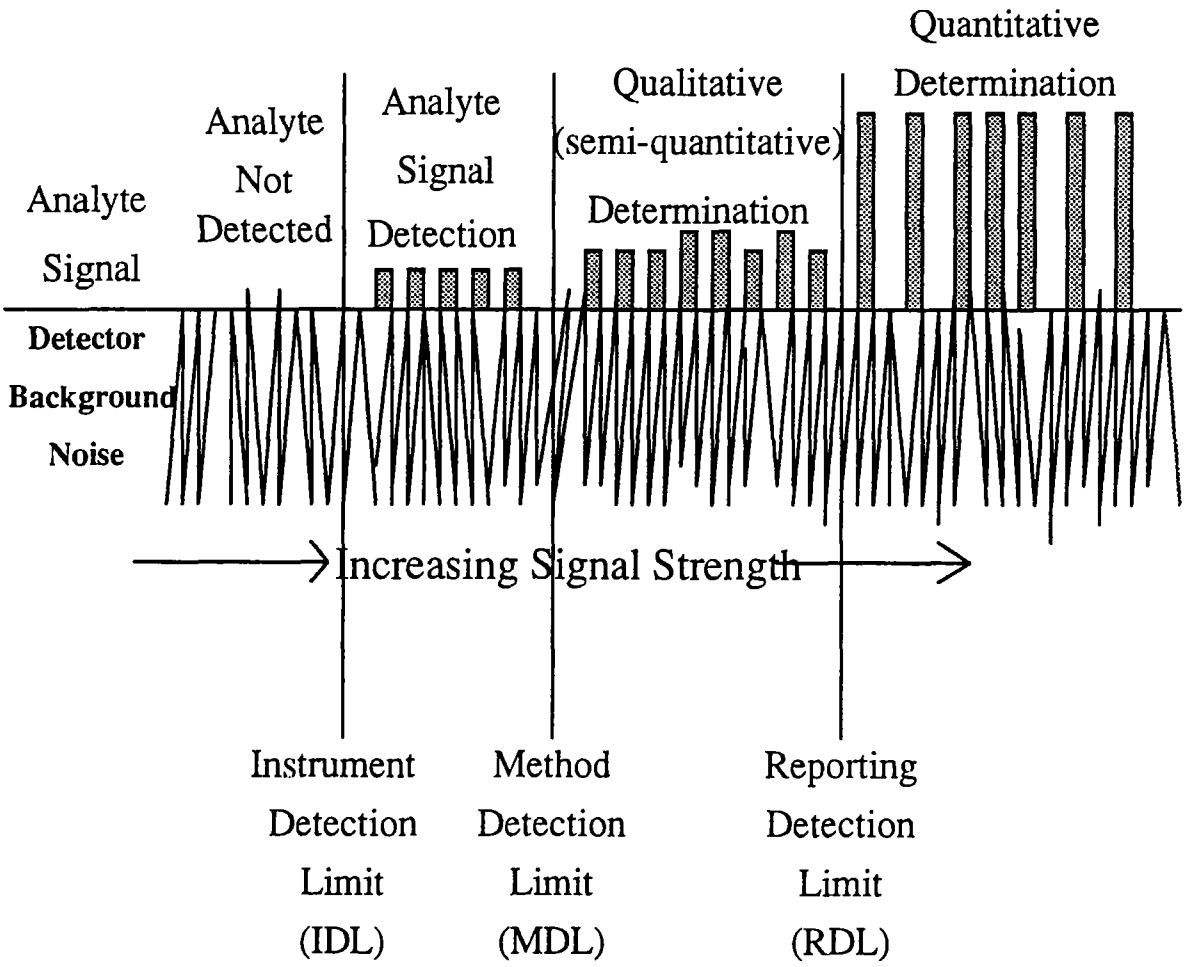


Figure 8

3.9.3 Procedures for Accepting New Work/Tests

3.9.3.1 New Test Requests

Client Services will forward a request for new analyses to the methods development group within each laboratory area where the request will be formally processed. Evaluation of the request will include the suitability of the analyte for quantitation, availability of existing test methods, instrumentation, standard materials, etc. The area manager, technical director, and/or group leader will provide a prompt response to client services to ensure that client needs can be addressed.

All newly developed methods are reviewed by the laboratory Technical Director and must comply with all requirements outlined in sections 3.8.2.1 and 3.8.2.2 of this manual.

3.9.3.2 Method Development and Approval

A brief summary of Development Steps is as follows:
The linearity of analytical response must be demonstrated in the range of interest to the client. For organics, the suitability of a compound for chromatography must be determined. If extraction must be performed, the efficiency of that process for a given analyte must be demonstrated. Instrument detection limits (IDLs) must be arrived at, and method detection limit (MDL) studies must be performed. A check for interferences from other analytes must be done. Finally, formal approval must be obtained by laboratory management and by regulatory agencies involved.

3.10 LABORATORY FACILITIES, EQUIPMENT AND SUPPLIES

3.10.1 Physical Plant

3.10.1.1 Laboratory Demographics

The TriMatrix Grand Rapids laboratory is located at 5555 Glenwood Hills Parkway, SE, Grand Rapids, Michigan. The laboratory was constructed in 1988 and was designed to accommodate the strict analytical testing requirements in today's marketplace.

The 12,900 square foot facility was designed by the TriMatrix laboratory staff in correlation with our own design architects (Figure 9). Special attention was paid to sample preparation areas and in the segregation of non-compatible areas such as GC/MS semi-volatiles and volatile organics. A breakdown of each general area of analysis and the space allocated is as follows:

Analysis	Space Allotted, Ft^2
Wet Chemistry/Microbiology	Approx. 2000
Atomic Absorption/Emission	Approx. 2000
Volatile Organics	Approx. 2400
Semi-Volatile Organics	Approx. 1500
Sample Processing & Storage	Approx. 1500
Administrative Offices	Approx. 2500
Organic Pretreatment	Approx. 1000

Sample storage areas include a large walk-in cooler with satellite commercial storage refrigerators for the segregation of volatile organic samples.

Managers, Group Leaders, Chemists, Project Chemists and the General Staff utilize quiet office areas for data reporting, validation and reporting.

The TriMatrix laboratory is equipped with a complete Laboratory Information Management System (LIMS) which was specially

developed in-house to accommodate the specific needs of the laboratory and our clients.

Under the direction of the Laboratory Manager, TriMatrix is organized into the following operating areas and support services.

Administrative and Management Operations

Laboratory Administration
Client Services
Marketing/Sales
Project Management
Health and Safety
Quality Assurance
Computer Services

Analytical Operations
Inorganic Laboratory
- Metals Laboratory
- Non-Metals

Organic Laboratory
- Volatile Organic Laboratory
- Semi-Volatile Organic Laboratory
- Organic Extraction Laboratory

(Refer to Section 3.3.7 for Laboratory Organization Chart)

3.10.1.2 Reagent Water Systems

The TriMatrix Grand Rapids Laboratory utilizes a series of water treatment systems to obtain both ASTM Type I and Type II quality water.

The system begins with a supply line from the Grand Rapids Potable Water Distribution System. This source water is then passed through an activated carbon bed filter (to remove residual chlorine) softened prior to introduction to a reverse osmosis (RO) system. The RO process removes approximately 90% of the dissolved constituents. After the RO process, the treated water is stored in a 120 gallon storage tank. This capacity tank provides an ample supply of water for a full day of laboratory operations. The RO water is then distributed to the laboratory through a mechanical pump through two mixed bed deionizing canisters. This supply now meets the requirements of ASTM Type II, and is utilized for glassware cleaning and as a feed water to a variety of polishing systems.

The polishing systems in place are comprised of several distillation units and a Milli-Q 4 Bowl System. Distilled RO-Deionized water is used primarily for BOD and metals analyses. Mill-Q water, which is equivalent to an ASTM Type I designation is used primarily for the preparation of standard solutions and reagents.

Each water system is monitored on a daily basis, checked against pre-defined acceptance criteria and recorded for documentation purposes.

Responsibility for monitoring the TriMatrix reagent water systems distributed between each laboratory area, is coordinated through the Quality Assurance Manager.

3.10.1.3 Glassware

For an analysis, glassware is selected which will provide the accuracy for that particular analytical procedure. TriMatrix purchases pipets, burets, and volumetric flasks, usually Class A, to meet the required accuracy. A standard operating procedure for cleaning glassware for each type of analysis is followed. Cleaning of glassware is performed according to the analysis being conducted, and the sample matrix

involved, but certain general rules do apply. Use hot water to wash water-soluble substances. Use detergent, dichromate solution, organic solvent, nitric acid, or aqua regia to remove other materials according to the specific glassware cleaning procedures. Avoid using detergents on glassware to be used for phosphate determinations. Use ammonia-free water for ammonia and kjeldahl nitrogen analyses. In all analyses, it is advisable to rinse glassware with tap water followed by deionized water immediately after use; matrix materials which are allowed to dry on glassware are more difficult to remove by washing.

3.10.1.4 Compressed Air

Compressed air must be free of dirt, water, and oil. For compressed air purchased from vendors, specification shall be high purity grade (breathing air). For compressed air produced in the laboratory, use filters at the compressor to keep water from moving into the compressed air delivery system. For certain instruments, such as gas chromatographs and atomic absorption spectrophotometers, install filters at the instrument to remove oil from the compressed air supply.

3.10.1.5 Ventilation Systems

Each laboratory operating area has its own separate air-handling system. In addition, the organic area has separate systems for the volatiles and semi-volatiles instrument rooms. Positive pressure is maintained on the instrument rooms at all times. The air-handling systems, which control heating, cooling and humidity, also maintain maximum cfm air turnover.

3.10.1.6 Electrical Services

The electrical system at the TriMatrix Laboratory was designed and installed specifically for use in a laboratory environment. Special attention was paid to instrument requirements and the isolation of

separate lines for critical applications like GC, GC/MS, atomic absorption and automated analyzers.

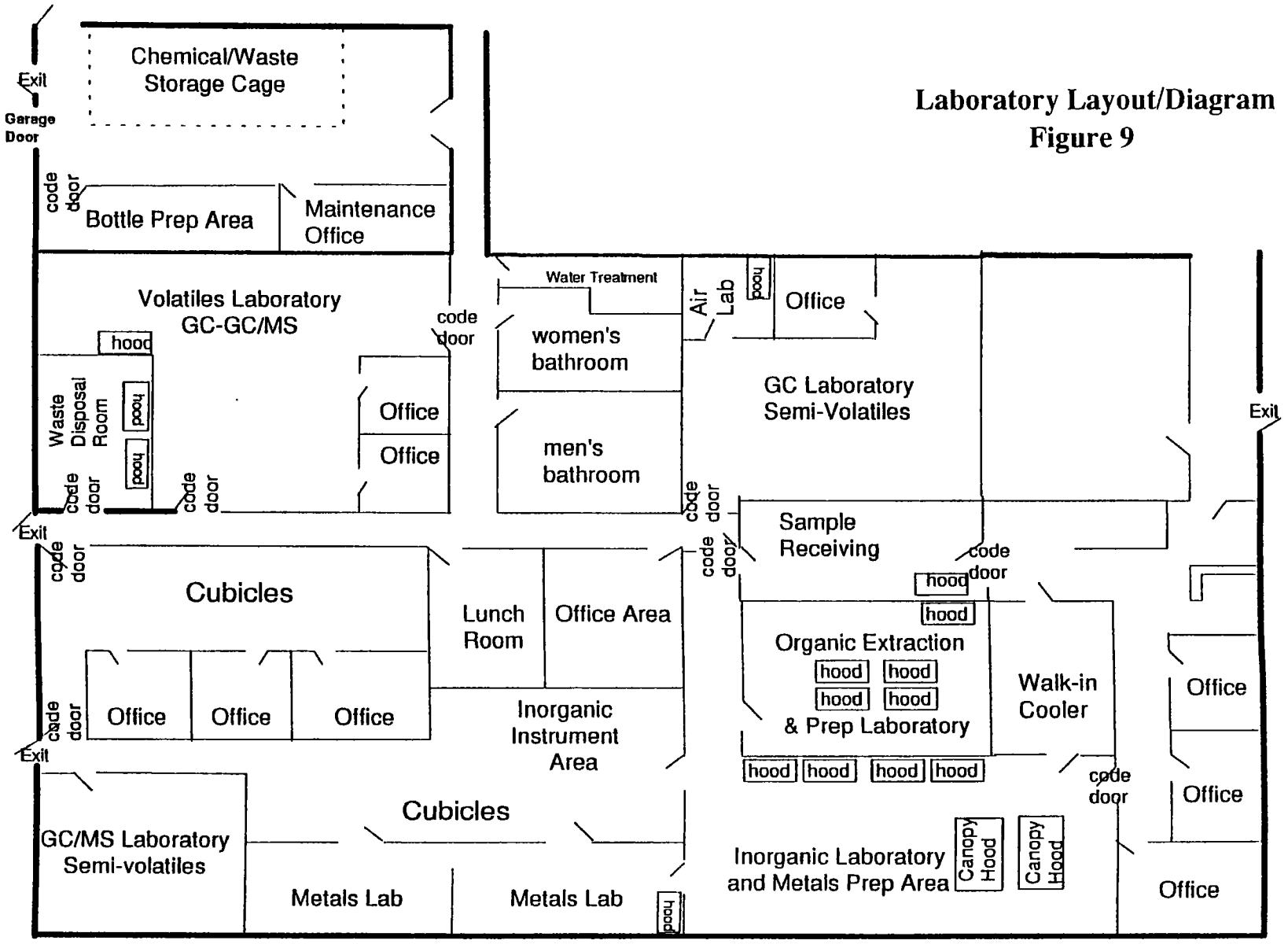
All laboratory benches, hoods and work areas were designed to accommodate a large number of laboratory applications, such as distillations, digestions and extractions.

Surge-protection devices are in place for all laboratory computing equipment. The main Laboratory Information Management System is also protected by a programmable uninterrupted power supply (UPS) device. This UPS allows for a sequence shut-down of the LIMS system during a power failure. This sequenced shut-down provides excellent protection of the LIMS database during a power outage.

3.10.1.7 Laboratory Layout (Diagram)

Refer to Attached Floor Plan Figure 9

Laboratory Layout/Diagram
Figure 9



3.10.2 Equipment Procurement

For an environmental testing laboratory where trace analyses are routinely performed, certain specifications for laboratory equipment are critical to quality. Instrumentation, balances, glassware, water baths, etc. must have the accuracy required for particular analytical procedures. The Laboratory Area Manager in conjunction with the Laboratory Manager and the Technical Director are responsible for determining the required specifications before equipment is procured. The analytical specifications are based on a detailed review of the test methods.

3.10.3 Equipment Management/Maintenance/Inventory

Adequacy of equipment for its intended purpose must be verified before use. A sufficient inventory of equipment is to be maintained so that testing delays are not incurred due to shortages. A stock count is kept on each item that automatically triggers a reorder point and avoids expensive express charges for rush orders. Service is performed on equipment on a scheduled basis. A stock of critical equipment spare parts which are known to wear out regularly are maintained.

Maintenance log books are kept so that the maintenance requirements of each major equipment item can be determined over a time interval.

A complete listing of Laboratory Equipment is presented in Appendix B of this manual.

3.10.4 Chemical Procurement and Inventory

3.10.4.1 Reagents, Solvents, and Gases

The purchasing of reagents, solvents, and gases must be carefully controlled through the purchase order system so that a minimum quality level is maintained for these raw materials in the testing process. The Managers of each laboratory operating area along with

the Quality Assurance Manager will define the suitable grades of materials. The Area Manager, or his designate will verify upon receipt that the incoming materials meet requirements. Selected types of incoming reagents, solvents, and gases are assigned an identification number for traceability in laboratory testing records (Note: the manufacturer's lot number may be used if it meets the traceability requirements and is pre-printed on each container label.) Material certificates of analysis are to be provided by the vendor and stored in laboratory files. Each area manager, or his designate, is to oversee proper storage and removal of reagents, solvents, and gases when their shelf-life has expired.

Reagents, solvents, and gases are available from vendors in a broad range of purity - from technical to ultrapure grade. The type of analysis, as well as the sensitivity and specificity of the method must be considered in choosing a grade of material. The analytical reagent (AR) grade is suitable for most inorganic analyses. Trace organic analyses frequently require a special ultrapure grade. AR grade is the minimum for reagents and solvents used in organic analysis. The absence of certain impurities is required for some GC detectors - notably sulfur and phosphorus in the FID detector. Trace metals analyses including atomic emission and atomic absorption spectroscopy usually require spectro-quality reagents and solvents, although AR grade may be suitable in some cases. In sample cleanup procedures involving florisil, silica gel, and alumina as absorbents, these substances must be checked for interfering components when preactivated according to the analytical method. Compressed gases are available in various purities, usually expressed as a percent (e.g. 99.999) along with a certificate of analysis showing the maximum contaminant level. Gases should be filtered in the laboratory delivery lines to remove moisture, oil, and other contaminants. Refer to the analytical methods and the instrument manufacturer's operating manual for gas purity requirements. In the use of all reagents,

solvents, and gases the analyst must verify the purity requirements as well as the grade of the material on hand before beginning an analysis.

Shelf life of chemicals purchased from vendors is based on the following guidelines (unless otherwise specified by the manufacturer or derived from other information):

Inorganics

Liquids - 5 years
Solids - 5 years
Compressed Gases - 6 months

Organics

Liquids - 2 years
Solids - 5 years
Compressed Gases - 6 months
Ethers generally are to have an expiration date of 34 days due to the potential for peroxide formation.

3.10.4.2 Certified Standards

The purity and traceability of standards used in analytical processes is crucial to the quality of data being generated. Only high quality standards certified by established vendors are to be utilized. Calibration reference standards must be of the designated purity required for a particular analysis. Obtain primary reference standards and standard solutions from the National Institute of Standards and Technology (NIST), the USEPA Repository, or other high-quality commercial sources.

Chemical reference standards obtained from established commercial vendors should be traceable to NIST. Maintain a log of all standards and reference solutions received from vendors. Identify the supplier, lot number, concentration, date received, and any dilution or preparation performed after receipt. Write the date received on the

standard container label. Each container of incoming standards is to be assigned an identification number for traceability in laboratory records (Note: the manufacturer's lot number may be used if it meets the traceability requirements and is pre-printed on each container label).

Stock and working standards must have a label describing the compound name, concentration, date prepared, name of preparer, and expiration date.

Validate all standards prior to use by checking the purity against a standard obtained from a different commercial source. Evaluate stock and working standards periodically for signs of deterioration (i.e., precipitates, change in color, change in concentration).

During analytical operations, use standards from different vendors to cross-check each other. For example, perform an initial calibration with a standard from one commercial source and the continuing calibration verification standard from a different vendor.

In a situation where standard reference materials are not commercially available for an analysis, use compounds of certified high purity to prepare calibration standards and thoroughly document the procedure.

Utilize a computer database to maintain records of standards, showing the information in the receiving log described above as well as expiration dates, stock and working standards prepared from a particular vendor shipment, and other pertinent information for traceability and reordering.

Vendor expiration dates for purchased stock standards are not to be exceeded. Establish expiration dates for working standards based on guidelines in the analytical method, generally 6 months for working, and 1 year for stock standards.

4.0 QUALITY CONTROL

4.1 DOCUMENT CONTROL AND MAINTENANCE

4.1.1 Traceability of Measurements/Documentation Requirements

The purpose of a properly designed and implemented documentation protocol is to assure that after the issuance of an analytical report, all information presented can be fully traced back to its point of origin. This documentation system must also provide for traceability of non-reported information that provides supporting value to the analytical test results. These items include but are not limited to: stock standard records, test calibration records, data reduction and validation activities, sample custody, facilities monitoring and final data reporting.

A more detailed review of the documentation procedure and traceability of information is presented below.

4.1.2 Paperwork/Information Flow

As displayed in Figure 11, the flow of documents is the same for general routine work as it is for samples under strict chain-of-custody (COC). The general axiom is that a COC procedure will fail without a pre-existing scheme of rigid documentation control available as a routine measure. The records trace can provide for the following:

- Answers to questions of analytical integrity for results which have been reported for 2 days or 2 years.
- Assistance in finding and solving random and systematic problems.
- Assistance in preventing long term degradation of the analytical process.
- Assistance in ensuring continuity of analytical effort despite personnel and mechanical changes.

The following subsections identifies and describes the procedures and any corresponding documents that are generated during the course of any project/submittal through the laboratory.

4.1.2.1 Project Initiation

All samples or sample groups which enter into the TriMatrix analytical process, must be accompanied by the appropriate documentation. This documentation is necessary to define the analytical goals and project objectives. These documents will include one of the following: for larger projects with federal or state oversight, a quality assurance project plan-QAPjP is written; an TriMatrix laboratory project request form. In conjunction with a QAPjP or an TriMatrix Project Initiation Form, an inventory of required sample containers must be prepared for each sampling event. This inventory is prepared on an TriMatrix master bottle packing list. Examples of the TriMatrix forms are presented in Figures 12 and 13.

The TriMatrix project request form is utilized for both the development of project objectives and in the creation of laboratory price quotations.

The TriMatrix laboratory project initiation form is completed to assist the implementation of the project initiation step within the TriMatrix LIMS system.

All projects initiated or developed within LIMS, are validated by means of a project report (see Figure 14). This report presents all project information including tests requested, methods, custom fraction lists, special reporting limits and special quality control limits if required. All information depicted on this report is validated by the appropriate project chemist prior to sample login. Modifications to project information can be accomplished after login through the project maintenance module in LIMS.

All documents created during the project initiation phase are maintained and archived to the client filing system.

4.1.2.2 Sample Receipt/Examination

Immediately upon receipt of a sample shipment or delivery group at the TriMatrix laboratory, the sample coordinator (SC) will examine the shipping container to ascertain and document the condition of the samples. This examination is performed and documented on three different TriMatrix reports. These three reports are designed to communicate not only the conditions of the shipping containers during sample receipt, but also any problems associated with a particular sample delivery group.

When all samples from a sample delivery group have been removed from the shipping containers and examined against the external QC forms, only then will the SC sign-off for receipt the laboratory. In many cases, COC form is presented to the sample courier for their record of sample delivery to the laboratory.

The "Cooler Receipt Form" (see attached Figure 15, is utilized for all internal laboratory chain-of-custody projects as well as all projects requiring reporting levels of 4, 5, or 6.

The SC will perform a series of measurements to check and document each coolers temperature, and the pH of all chemically preserved sample containers with the exception of solid matrices. Measurements performed during these steps are recorded on the TriMatrix '"Sample Preservation Log Form" (see attached example in Figure 16).

The third form or the "Problem Submittal Report (see attached figure 17) is issued for all sample delivery groups that have cited deficiencies or non-conformance's from stated sample delivery, preservation or custody policies.

If a problem is encountered with a sample delivery group, an TriMatrix Nonconformance Report is issued with the problem submittal report to the appropriate project chemist for corrective actions.

The sample coordinator, having opened the shipping containers and examined all the samples, will next verify the existence of an initiated project within the TriMatrix LIMS system. In most cases, the representative project chemist will provide the sample coordinator with a "project report", detailing the project information including the tests required for all samples. Any discrepancies in the requested tests and the sample containers received is forwarded to the project chemist for corrective actions.

TriMatrix Laboratories, Inc.
Document - Benchsheets/Client Report
Flow Diagram

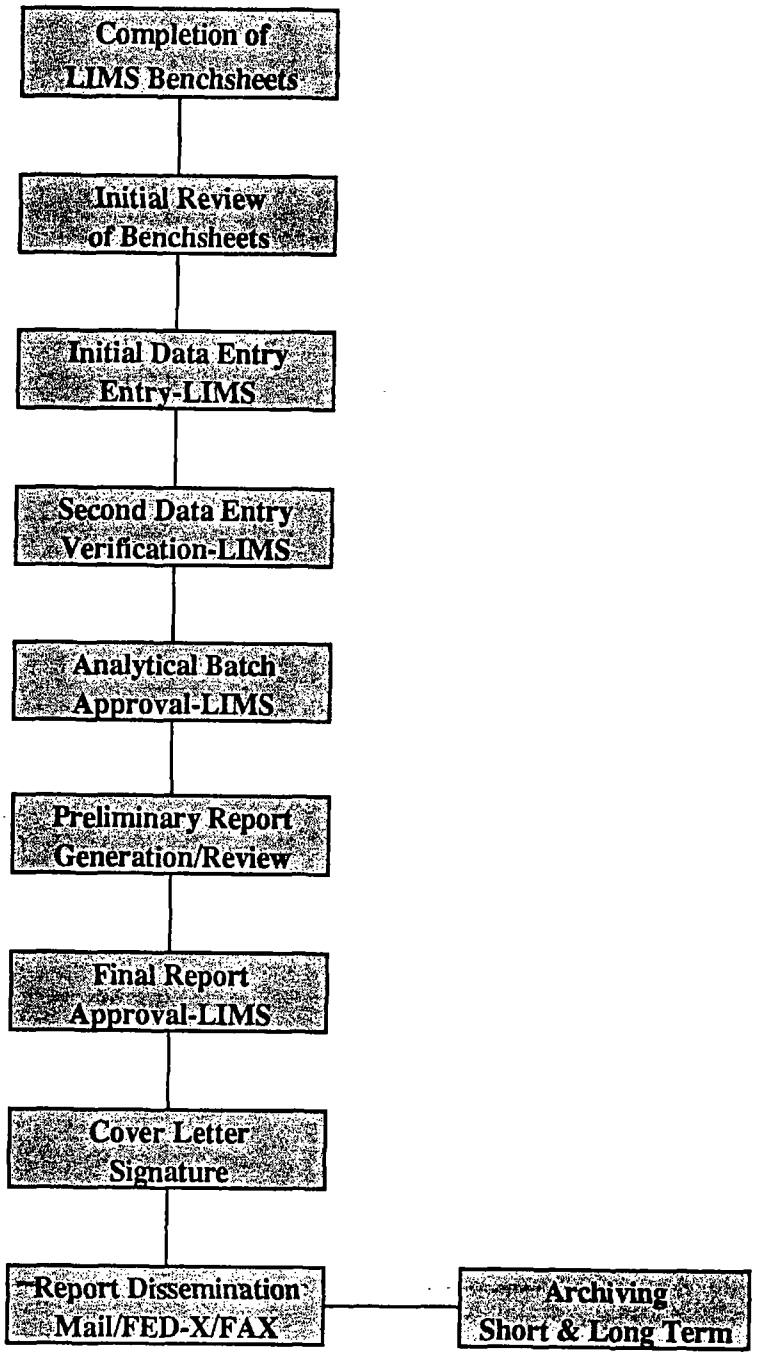


FIGURE11.XLS

Figure 11

TriMatrix Laboratories, Inc.

PROJECT INFORMATION FORM

REQUEST FOR: ___WORK ____QUOTATION
TODAY'S DATE: _____/_____/_____
DATE NEEDED: _____/_____/_____

Client Information:

1. Client Name:_____
2. Project Name:(as it is to appear on the report):_____

3. TriMatrix Project Number:_____

3a. TriMatrix Project Manager:_____ TriMatrix Contact:_____
TriMatrix Office:_____ TriMatrix Office:_____
Telephone Number:_____ Telephone Number:_____
FAX Number:_____ FAX Number:_____

Project Information:

1. Expected Date of Sampling:___/___/___ Bottles needed by:___/___/___
_____ to be picked up by TriMatrix Personnel.
_____ to be shipped to: _____
_____
_____

1b. Special Instructions for Sample bottle and cooler preparation:
_____

2. Project Frequency:
_____ One Time Event _____ Weekly Event _____ Semi-Annual Event
_____ Monthly Event _____ Quarterly Event _____ Annual Event
_____ Other - (Please explain):_____
3. Projected date samples would arrive in the laboratory:____/_____/_____
4. Number of Samples: _____Matrix Type(s):_____
4a. Parameters Required/Detection Limits are:(attach separate sheet if needed)

Parameter (s)	Detection Limit/units

Figure 12

TriMatrix Laboratories, Inc.

4b. Special Methods Required and/or program (permit) requirements:

_____

_____

_____

_____

5. Will there be any field blanks? _____ YES _____ NO
(Please indicate in Section 4a (above) what parameters are to be analyzed.)

6. Will there be any trip blanks needed? _____ YES _____ NO
(Please indicate in Section 4a (above) what parameters are to be analyzed.)

7. Potential Hazards associated with the samples are:_____

8. Has the client that any hazardous samples will be returned to them?
_____ YES_____ NO

9. Disposal of non-hazardous samples will take place thirty (30) days after the report mailing unless otherwise requested. The cost for storage past this time is $??.?? per sample.

10. Is Strict Chain of Custody needed for this project?
_____ YES Please Specify special needs:_____
_____ NO (The laboratory will use its standard protocol.)

11. Analytical turnaround required:
_____ Laboratory Standard (3 Weeks) _____ Two (2) weeks
_____ One (1) week _____ Other:(Specify)_____

Reporting Information

1. Please indicate below the type of Quality Control Reporting that you will need for your project:

Level of QC Reporting	**Type of Information you will receive**
_____ Level 1	Cover Letter, Invoice, Report and SDQ*
_____ Level 2	Level 1 + Batch Method Quality Control Data
_____ Level 3	Level 2 + Matrix Specific Quality Control Data
_____ Level 4	Level 3+ Raw Data Package
_____ Level 5	Level 3 + CLP Forms Package
_____ Level 6	IRPIMS Electronic Deliverables Package

*Statement of Data Qualifications Report

2. Please indicate any special reporting format(s) that are needed for this project.

_____

3. Are disk deliverables needed? _____ YES _____ NO

3a. If disk deliverables are needed, what type of format? (i.e.:Lotus, Excel, etc.) Please specify:_____

4. Please indicate the report distribution of the final report:_____

5. Other Information the laboratory needs:_____

Please sign and date below and return to the appropriate project chemist for this project. Please keep a copy for your records.

Project Manager: _____

Date: _____/_____/_____

Figure 12

TriMatrix
Laboratories, Inc. 5555 Glenwood Hills Parkway SE • PO Box 888692 • Grand Rapids, MI 49588-8692 (616) 975-4500

Client: _____

Project: _____

Project Manager: _____ Contact: _____ Project No.: _____

Bottles Requested on: _____ Date Due to Client: _____ Time Due: _____

Bottles will be Shipped / Picked Up _____

If Shipped: ☐ Overnight ☐ 2nd Day Air ☐ Standard ☐ Best Way
☐ Other _____

Ship Bottles to: _____

Frequency: ☐ One Time ☐ Quarterly ☐ Others _____
☐ Weekly ☐ Semi Annual
☐ Monthly ☐ Annual

Prepare Bottles for:

Months ☐ Jan ☐ Feb ☐ Mar ☐ April ☐ May ☐ June
☐ July ☐ Aug ☐ Sep ☐ Oct ☐ Nov ☐ Dec

Weeks: ☐ 1 ☐ 2 ☐ 3 ☐ 4 ☐ 5

Day: ☐ M ☐ T ☐ W ☐ TH ☐ F

Expiration Date: _____

COMMENTS

_____

_____

_____

_____

_____

_____

_____

_____

Bottle Prep Use Only _____ **Date Shipped:** _____

Coolers Assigned _____ **Tracking No.:** _____

_____ **Courier ID:** _____

Figure 13

Client Name: _____

For any questions regarding these bottles, contact Barry Huizenga, the Project Chemist for this submittal.

Sample Inventory and Master Bottle Packing List

Sample Locations	Sample Sub-Portions-Preservative and Tagging Codes																								
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25
Field Filtering Required Yes / No																									

NO.	DESCRIPTION	PRESERVATIVE	TAG COLOR
	Water		
1a	40 ml Vial for Purgeable Organics	1+1 HCL Cool to 4° C	Yellow
1b	40 ml Vial for Purgeable Organics	Cool to 4° C	Yellow & Black Stripe
2	1000 ml Amber Glass Non Purgeable Organics	Cool to 4° C	Salmon
3	125 250 500 1000 ml Plastic - Non Preserved	Cool to 4° C	Green
4	125 250 500 1000 ml Plastic - Nutrients	pH < 2.0 w/H_2SO_4	Dark Blue
5	500 1000 ml Amber Plastic - Cyanides	pH to > 12 w/NaOH	Light Blue
6	125 250 500 1000 ml Plastic - Metals	pH to <2 w/HNO_3	Red
7	1000 ml Glass - Oil & Grease / TPH	pH to <2 w/H_2SO_4	Dark Blue
8	125 ml Bottle Bacteria	Cool to 4° C	White Label
9	500 ml Glass - Sulfide	0.5 ml Zinc Acetate + 0.5 ml NaOH to pH >9	Light Green
10	500 ml Amber Glass - TOX	pH to < 2 w/H_2SO_4 Cool to 4° C	Lilac
11	40 ml Amber Glass - TOC	pH to < 2 w/H_2SO_4 Cool to 4° C	Pink
12	2000 ml Plastic - Radiological	pH to < 2 w/HNO_3	Gray
13	500 ml Amber Glass - Phenols	pH to < 2 w/H_2SO_4	Brown
14	250 ml Amber Glass - Formaldehyde	Cool to 4° C	Orange
15	125 250 500 1000 ml Plastic - Dissolved Metals	pH to < 2 w/HNO_3	Red & White Stripe
	Soil / Non-Aqueous		
16	125 250 500 1000 ml Wide Mouth Plastic	Cool to 4° C	White
17	125 250 500 1000 ml Wide Mouth Amber Glass	Cool to 4° C	Manilla
18	125 ml Vial for Purgeable Organics in Soil	Cool to 4° C	Light Yellow
19	125 ml Vial for TCLP Volatiles	Cool to 4° C	Yellow & Black Stripe
20	125 ml Wide Mouth Plastic - % Solids	Cool to 4° C	Yellow & White Stripe
	Other Water / Soil / Non-Aqueous		
21			
22			

WATER SAMPLING

SOIL SAMPLING

Figure 13

01-MAR-1995 PROJECT REPORT - ONE

NUMBER 29454
DESCRIPTION Water Pollution Study Semi-Annual PE
CLIENT 29454
PROJ. CHEMIST 1991
CONTACT Kriscunas, Douglas
PHONE (616) 942-9600 EXT.
ENTERED BY DEK
ENTERED ON 05-SEP-1991
TURN AROUND 21 DAYS
CHAIN OF CUSTODY E
QC REPORTING H
CASE NARR FLAG Y
BOTTLE HANDLING 3
BOTTLE CARRIER DEK
HOLD DAYS NON HAZARDOUS LAB DISPOSAL
DISPOSAL 1

HAZARDS: 1- FLAMMABILITY; 1- REACTIVITY; 1- CONTACT; - HEALTH

SAFETY
CCS NUMBER CCS MANAGER
PO NUMBER CONTRACT 05-SEP-199
EXPIRATION DATE 180
SUBMITAL FREQUENCY 2 DAYS SUBMITTALS PER YEAR 20
FACTOR %U SAMPLES PER SUBMITTAL Water
PRICE CODE Y

BUSINESS OFFICE PROJECT NAME Qc Rept Flag Y

NARRATIVE:

Figure 14

01-[illegible]995 PROJECT REPORT - ONE

NUMBER 29454
DESCRIPTION Water Pollution Study Semi-Annual PE
CLIENT 29454

	QTY	ACTIVATE	INACTIVATE
PCB'S in Oil 1 & 2	2		

PCB'S USEPA-8080 SCAN GC/ECD/P&P/WST USEPA-8080 WASTE ----- N mg/kg N

		Common Name	Detect Limit	Parm Num	Parm Var	Meth Num	Meth Var
GC/ECD/P&P/WST	-----	PCB-1016	0.5	502	1	39	25
GC/ECD/P&P/WST	-----	PCB-1016	0.5	502	1	39	25
GC/ECD/P&P/WST	-----	PCB-1221	0.5	502	1	39	25
GC/ECD/P&P/WST	-----	PCB-1221	0.5	502	1	39	25
GC/ECD/P&P/WST	-----	PCB-1232	0.5	502	1	39	25
GC/ECD/P&P/WST	-----	PCB-1232	0.5	502	1	39	25
GC/ECD/P&P/WST	-----	PCB-1242	0.5	502	1	39	25
GC/ECD/P&P/WST	-----	PCB-1242	0.5	502	1	39	25
GC/ECD/P&P/WST	-----	PCB-1248	0.5	502	1	39	25
GC/ECD/P&P/WST	-----	PCB-1248	0.5	502	1	39	25
GC/ECD/P&P/WST	-----	PCB-1254	0.5	502	1	39	25
GC/ECD/P&P/WST	-----	PCB-1254	0.5	502	1	39	25
GC/ECD/P&P/WST	-----	PCB-1260	0.5	502	1	39	25
GC/ECD/P&P/WST	-----	PCB-1260	0.5	502	1	39	25

Pesticides 1 & 2 2

PEST-608 USEPA PE'S GC/ECD/P&P/WTR USEPA-608 WTR 1.0 N ug/l N

		Common Name	Detect Limit	Parm Num	Parm Var	Meth Num	Meth Var
GC/ECD/P&P/WTR	1.0	ALDRIN	0.01	503	5	39	11
GC/ECD/P&P/WTR	1.0	ALDRIN	0.01	503	5	39	11
GC/ECD/P&P/WTR	1.0	4,4'-DDD	0.02	503	5	39	11
GC/ECD/P&P/WTR	1.0	4,4'-DDD	0.02	503	5	39	11
GC/ECD/P&P/WTR	1.0	4,4'-DDE	0.02	503	5	39	11
GC/ECD/P&P/WTR	1.0	4,4'-DDE	0.02	503	5	39	11
GC/ECD/P&P/WTR	1.0	4,4'-DDT	0.02	503	5	39	11
GC/ECD/P&P/WTR	1.0	4,4'-DDT	0.02	503	5	39	11
GC/ECD/P&P/WTR	1.0	DIELDRIN	0.01	503	5	39	11
GC/ECD/P&P/WTR	1.0	DIELDRIN	0.01	503	5	39	11
GC/ECD/P&P/WTR	1.0	HEPTACHLOR	0.01	503	5	39	11
GC/ECD/P&P/WTR	1.0	HEPTACHLOR	0.01	503	5	39	11
GC/ECD/P&P/WTR	1.0	HEPTACHLOR EPOXIDE	0.01	503	5	39	11
GC/ECD/P&P/WTR	1.0	HEPTACHLOR EPOXIDE	0.01	503	5	39	11

Figure 14

TriMatrix Laboratories, Inc.

Cooler Receipt Form
Part 1

Cooler #_____

Project: _____ Date Received: _____ Project Submittal No. _____

Use other side of this form to note further details concerning check-in problems and to specify and describe any action(s) regarding the resolution(s) of problems. If shipment was accepted and if requested. Note on back the address where the empty cooler was returned and likewise if the shipment was rejected.

A. **Preliminary Examination Phase:** Date cooler was opened: _____

by (print) _____ (sign) _____

1. Were custody seals on outside of cooler?.....YES NO

   If YES, how many & where: _____

   Date & Signature correct? If YES, seal date: _____, name:_____.....YES NO

2. Were custody seals unbroken and intact at the date and time of arrival?.....YES NO

3. Were custody papers sealed in a plastic bag and taped inside to the lid?.....YES NO

4. Was project identifiable from custody papers? If YES, enter project name at the top of this form.....YES NO

5. Were custody papers filled out properly (ink, signed, etc)?.....YES NO

6. Did you sign custody papers in the appropriate place?.....YES NO

7. Did cooler come with a shipping slip (air bill, etc.)?.....YES NO

   If YES, attach & enter air bill or invoice number here:_____

8. Have designated person initial here to acknowledge receipt of cooler: _____ (date) _____

Cooler Receipt Form
Part 2

B. **Log-In Phase:** Date samples were logged-in: _____

by (print) _____ (sign) _____

9. Describe packing: _____

10. If required, was enough ice used? (temperature = _____) ..... YES NO

11. Were all bottles sealed in separate plastic bags? ..... YES NO

12. Did all bottles arrive unbroken and in good condition? ..... YES NO

13. Were all bottle labels complete (ID, date, time, signature, preservative, etc)? ..... YES NO

14. Did all bottle labels agree with custody papers? If NO, indicate discrepancies on back ..... YES NO

15. Were correct containers used for tests indicated? ..... YES NO

16. Were correct preservatives used when required? ..... YES NO

17. Was a sufficient amount of sample sent for tests indicated? ..... YES NO

18. Bubbles absent in VOA vials? If NO, list exceptions _____ ..... YES NO

19. Were the samples accepted for QA/QC testing? If NO, detail as instructed at top of this form ..... YES NO

20. Have designated person initial here to authorize further processing: _____ (date) _____

Figure 15

TriMatrix Laboratories, Inc.

5555 Glenwood Hills Parkway SE • PO Box 888692 • Grand Rapids, MI 49588-8692

Sample Log-In Log

Analysis Type (Preservation)	Container Description	Tag Color	Bottle #	pH Should be							
VOC'S (wtr.) (1+1 HCL)	40 ml vial glass	Yellow	1	<2*							
SVOC'S (wtr.) (Non-Pres.)	N.M. amber glass	Salmon	2	~7							
Inorganic (Non Pres.)	N.M. plastic	Green	3	~7							
Nutrients (H_2SO_4 Pres.)	N.M. plastic	Blue	4	<2							
Cyanides (NaOH Pres.)	N.M. plastic	Lt. Blue	5	>12							
Metals (HNO_3 Pres.)	N.M. plastic	Red	6	<2							
Oil & Grease (H_2SO_4 Pres.)	W.M. clear glass	Dk. Blue	7	<2							
Bacteria (Non-Pres.)	125 ml. bottle/bag	Brown	8	~7*							
Sulfide (NaOH + Zn Acetate Pres.)	N.M. amber glass	Lt. Green	9	>9							
TOX (H_2SO_4 Pres.)	N.W. amber glass	Lilac	10	<2*							
TOC (H_2SO_4 Pres.)	40 ml amber vial glass	Pink	11	<2*							
Radiological (HNO_3)	W.M. plastic	Gray	12	<2							
Phenols (H_2SO_4 Pres.)	N.M. amber glass	Brown	13	<2							
Formaldehyde (Non-Pres.)	N.M. amber glass	Orange	14	~7*							
Other Water	Other	-	15	*							
Soils (Non-Pres.)	W.M. plastic	White	16	*							
Soils (Non-Pres.)	W.M. amber glass	Manilla	17	-							
VOA's (Soil) (Non-Pres.)	125 ml. soil vial	Lt. Yellow	18	*							
TCLP VOA (Soil) (Non-Pres.)	125 ml. soil vial	Yellow & Black Stripe	19	*							
% Solids (Soil) (Non-Pres.)	125 ml. W.M. plastic	Yellow & White Stripe	20	*							
Other Soil/Non-Aqueous	Other	-	21	*							
		All Bottle Types Received (Y/N)									

Coolers Dropoff:

Cooler # _____	Temp. at receipt _____ °F	_____ °C	Cooler # _____	Temp. at receipt _____ °F	_____ °C		
Cooler # _____	Temp. at receipt _____ °F	_____ °C	Cooler # _____	Temp. at receipt _____ °F	_____ °C		
Cooler # _____	Temp. at receipt _____ °F	_____ °C	Cooler # _____	Temp. at receipt _____ °F	_____ °C		
Cooler # _____	Temp. at receipt _____ °F	_____ °C	Cooler # _____	Temp. at receipt _____ °F	_____ °C		

* Do not check pH at log-in
N.M. - Narrow mouth bottle type
W.M. - Wide n bottle type

Comments:

0018

Figure

TriMatrix Laboratories, Inc.

LABORATORY
PROBLEM SUBMITTAL REPORT

CLIENT: _____

RECEIPT: DATE:_____ TIME:_____ BY:_____

DEFICIENCIES CITED: NONE:_____

____ CUSTODY SEALS - ABSENT / NOT INTACT (IF REQUIRED)

____ SAMPLES RECEIVED - WITHOUT COOLANT.

____ CHAIN-OF-CUSTODY - ABSENT/INCOMPLETE

____ SAMPLES ABSENT - QUANTITY DOES NOT MATCH COC FORM

____ CHAIN-OF-CUSTODY - DOES NOT MATCH SAMPLE TAGS

____ SAMPLE TAGS / ABSENT

____ SAMPLE BOTTLES - BROKEN

____ SAMPLE BOTTLES - TYPES & QUANTITY DO NOT MATCH ANALYTICAL REQUIREMENTS.

____ SAMPLE PRESERVATIVES - INCORRECT FOR ANALYSIS

____ SAMPLE VOLUMES ____ INCORRECT FOR ANALYSIS ____ HEADSPACE IN VIALS

____ CORRECTIVE ACTIONS TAKEN AT LOGIN-SAMPLES WILL BE PROCESSED AS IS

_____

_____

____ SAMPLES PLACED ON HOLD UNTIL CORRECTIVE ACTIONS ARE ISSUED
DATE: _____ TIME: _____ BY: _____

____ CORRECTIVE ACTIONS REQUIRED:

_____

_____

DATE: _____ TIME: _____ BY: _____

ADDITIONAL COMMENTS:

_____

_____

Figure 17

4.1.2.3 Sample Log-In

During the sample log-in process, a series of documents and computer entry functions are completed and performed in an effort to document and validate this process. Entries are made on these documents and electronic forms to facilitate the log-in process. Documents and forms requiring entries from the sample coordinator include: the sample receiving log, LIMS sample receiving module, external COC forms, and the LIMS sample log-in module. In concert with these entry/documentation activities, several reports and logs as well as sample bottle tags are produced to validate the log-in activities. Examples of an Arrival Log and a LIMS generated sample bottle tag are presented in Figures 18 and 19.

Entries both in writing or electronic are validated by the recording or storage of the sample coordinator (SC) initials and date. Modifications to any entry is also documented, initialed and dated both on paper and within the TriMatrix LIMS system.

The exact protocols and operational conditions utilized for the sample log-in process can be found in the TriMatrix SOP for sample receiving/sample log-in. .

The SC will initiate a project or submittal file for each sample delivery group received. This file is labeled with the LIMS system generated project-submittal sequence, and will contain all documents associated with the sample receiving, sample log-in process. These documents will include: all external chain-of-custody forms, sample preservation records, shipping records, any client correspondence and a copy of the actual log for each submittal. Upon completion of the analytical process, the project file becomes part of the permanent record of each project.

UNCOMPLETED ANALYSIS WITH A SAMPLE LOGIN DATE) OR = 03-Mar-1995 AND (OR = 03-Mar-1995

MDNR-Gaylord District — TURNAROUND DAYS: 21 — PROJECT CHEMIST: NBK — QC TYPE: LV3 — COC: I
31914-10 — PROJECT MANAGER: — LAB DUE DATE: 21-MAR-1995 (TUE)
MDNR Case #1585 — Dockside — March 3, 1995 Submittal — CLIENT DUE DATE: 24-MAR-1995 (FRI)

SAMPLE #: 111724	Before	Resin				SAMPLED DATE: 01-MAR-1994 (TUE)
LEAD, TOTAL		USEPA-7421	WTR	0	!	
SAMPLE #: 111725	After	Resin				SAMPLED DATE: 01-MAR-1994 (TUE)
LEAD, TOTAL		USEPA-7421	WTR	0	!	
SAMPLE #: 111726	Influent					SAMPLED DATE: 01-MAR-1994 (TUE)
VOL'S BY 8021/SCAN-2 LIST		USEPA-8021	WTR	0	!	
SAMPLE #: 111727	Between					SAMPLED DATE: 01-MAR-1994 (TUE)
VOL'S BY 8021/SCAN-2 LIST		USEPA-8021	WTR	0	!	
SAMPLE #: 111728	Effluent					SAMPLED DATE: 01-MAR-1994 (TUE)
VOL'S BY 8021/SCAN-2 LIST		USEPA-8021	WTR	0	!	
SAMPLE #: 111729	Trip Blank					SAMPLED DATE: 03-MAR-1995 (FRI)
VOL'S BY 8021/SCAN-2 LIST		USEPA-8021	WTR	0	!	

SUBMITTAL NARRATIVES: VOA's in rack #489-W-14

PROJECT NARRATIVES:
** known as Case #1287 in October 1993.
** (was Dave Lindsay's project)
*The new LOE is McNamee Operational Services. Please send a
*copy of all reports to : McNamee Operational Services
* Attn. Mr. Jerry Randall
* 3131 S. State St.
* Ann Arbor, MI 48108
*THIS IS A 10TH OF THE MONTH PROJECT!!!

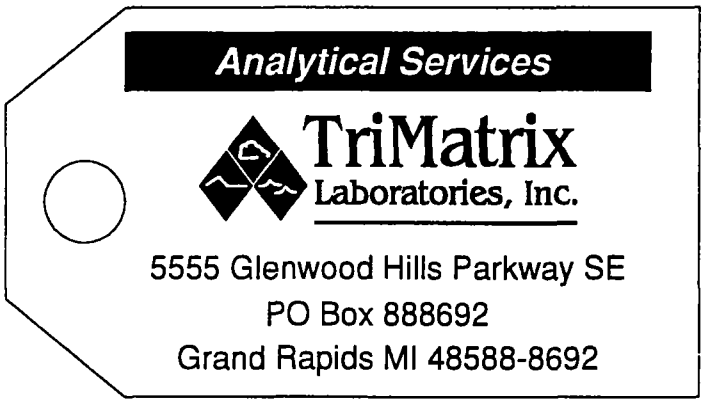
CLIENT NARRATIVES:
** always 307 detection limits unless
** the submittal or sample narratives state differently.
** this client narrative will always show up

Figure 18

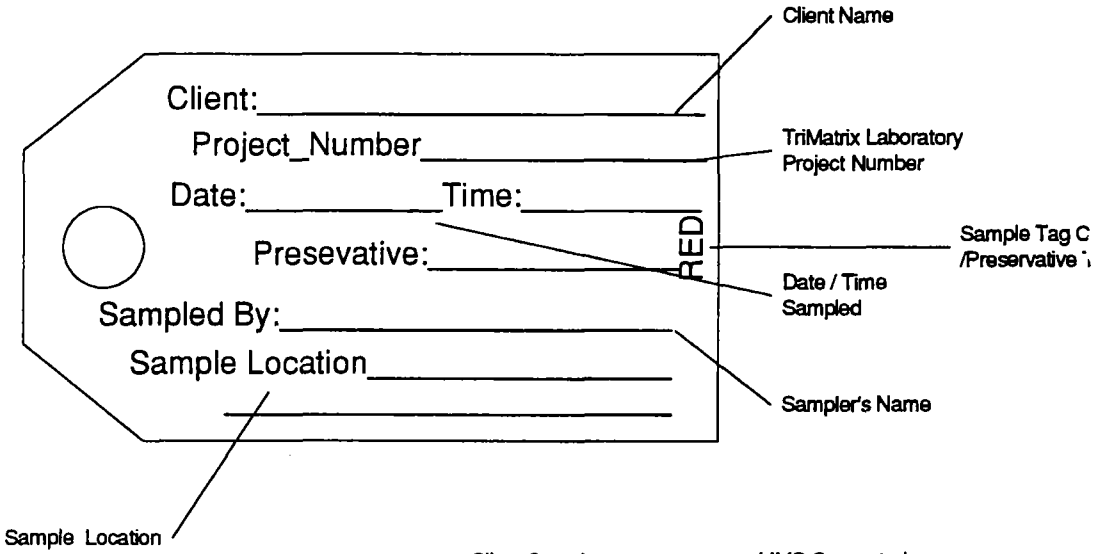
TriMatrix Laboratories, Inc.

Sample Bottle Tagging System

Tag Face



Tag Back



Tag Face with LIMS Generated Log-In I.D. Lable

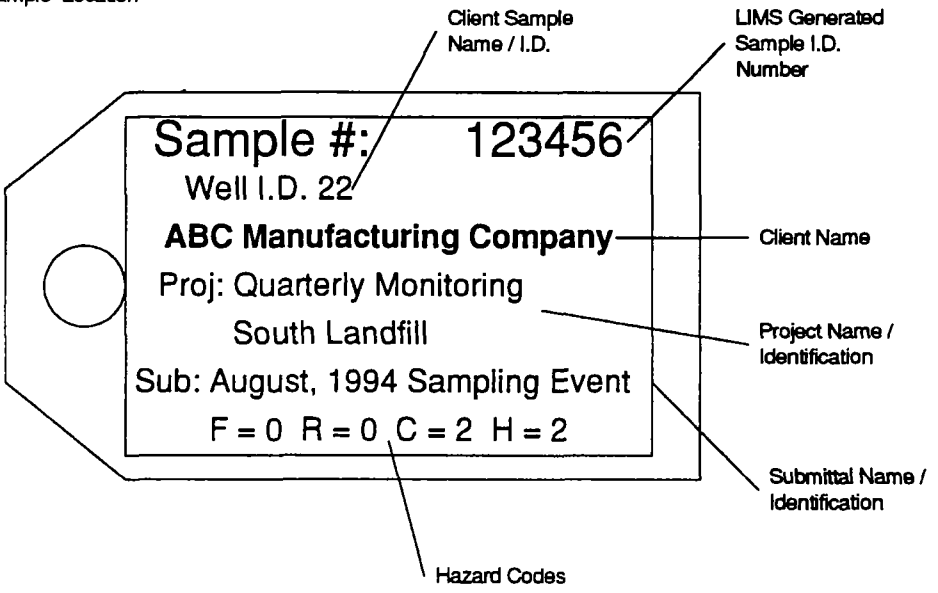


Figure 19

Parameter: NITROGEN, NITRATE (403.1) Method: NO3/AUTOAN/WTR (264.1) Ref. Cit.: USEPA-353.2 ODL: 0.05 Unit: mg/l

Client	Project / Submittal	QC	COC	Sample Description	First Sample	Sample Count	Anal hours	Hold-time exp. date	Lab date
USEPA Performance Evaluat	29063 Water Supply Analysis	LV2*		Ampul	105720	1	0.1	11-DEC 08:00	24-JAN

8 WS Study-35

Narratives	Entered	User	Text
Project:	24-OCT-90	DEK	****STUDY NOTES****
	24-OCT-90	DEK	(ORGANICS)
	24-OCT-90	DEK	SAMPLE #1 CONTAINS THE EIGHT REGULATED VOLATILE ORGANICS
	24-OCT-90	DEK	SAMPLE #2 CONTAINS NEW ADDITIONAL REGULATED VOLATILES.
	24-OCT-90	DEK	SAMPLE #3 CONTAINS UNKNOWN VOLATILE COMPOUNDS
	24-OCT-90	DEK	(METALS)
	24-OCT-90	DEK	THE CONCENTRATIONS IN SOME OF THE SAMPLES MAY BE BELOW OUR
	24-OCT-90	DEK	NORMAL FLAME/ICP LEVELS. FURNACE IS PREFERRED.
	24-OCT-90	DEK	.
	24-OCT-90	DEK	(GENERAL)
	24-OCT-90	DEK	ALL PROCEDURES USED IN PREPARING THE SAMPLES MUST BE ENTERED
	24-OCT-90	DEK	INTO THE QC PREP BOOK.
	24-OCT-90	DEK	IT IS VERY IMPORTANT THAT YOU READ "ALL" THE INFORMATION
	24-OCT-90	DEK	THAT IS SUPPLIED WITH THE AMPULS. THIS INFORMATION WILL IN
	24-OCT-90	DEK	MANY CAUSES PROVIDE YOU WITH THE ANALYTICAL RANGE OF THE
	24-OCT-90	DEK	CONCENTRATES.
	24-OCT-90	DEK	ALWAYS PREPARE AND ANALYZE A MPB = METHOD PREPARATION BLANK
	24-OCT-90	DEK	WITH EACH SET OF CONCENTRATES PREPARED.

Client	Project / Submittal	QC	COC	Sample Description	First Sample	Sample Count	Anal hours	Hold-time exp. date	Lab date
Keebler Company	29922 Mixing Water	LV1		3,8	108211	2	0.2	12-JAN 12:00	16-JAN

8 January 1995 Samples

Narratives	Entered	User	Text
Project:	27-DEC-91	RWH	LOG-IN: Please adjust due dates for each submittal to
	27-DEC-91	RWH	reflect a 5 working day turnaround to the client.
	27-DEC-91	RWH	The project is set up for 21 days so that no
	27-DEC-91	RWH	surcharge is automatically applied.

Figure 20

4.1.2.4 Worklists/Benchsheets

Upon completion of the sample receiving/sample log-in process, a series of worklists and selected benchsheets, are automatically printed for the new samples and analyses.

In the case of labile or short hold time parameter types, worklists are generated automatically each evening. These worklists are utilized for scheduling of short hold time tests and the production of any corresponding laboratory benchsheets. Most short hold parameters, i.e., BOD, hexavalent chromium, nitrite and nitrite nitrogen, turbidity, etc. are analyzed by the second and third shift staff members. An example of this worklist is presented in Figure 20.

Benchsheets for most routine tests are provided each evening or on demand through the TriMatrix LIMS system. Examples of selected benchsheets are presented in Figures 21 through 25.

The worklists and benchsheets produced by the TriMatrix LIMS system, are designed to provide the analyst with a gamut of important information. This information not only includes client/project specifications, but also provides an avenue for communication of test specifications, parameter expiration dates and times, formula's statistical control limits for all QC types and a uniquely designed form for recording calculating and reporting of analytical and QC data.

This up-front information enables the analyst to make vital decisions in their analytical scheme, and helps to minimize problems after samples are analyzed.

An example of a completed laboratory benchsheets are presented in Figures 26-27.

All laboratory benchsheets are dated and initialed at each review step. All approved benchsheet packages are scanned onto an optical disk, re-assembled and filed as part of the archiving process.

10-JAN-95 INORGANIC BENCHSHEET — BOD PAGE 1

Test #: 413. 1- 288.02
Parameter: BOD, (5-DAY)
Method: BOD/5-DAY/UW ODL: 1.0
Ref. Cit.: USEPA-405.1 Unit: mg/l

Batch: _____

Comments:

Instrument #:
Benchsheet ID: I25869
Owner:
Date Run:
Supervisor:
Est anal hrs: .5
Act anal hrs:
Samples in batch: 1
Stock std #:

BOD QUALITY CONTROL

	Blank	Bl Nut	Bl Seed	Bl Seed C	Seed	Seed	Seed	Seed C	Seed C	Seed C	LCS	LCS	LCS
SAMPLE #													
BOTTLE #													
INITIAL													
RESIDUAL													
SEED CORRECTION													
%/ML OF SAMPLE													
BOD MG/L													

LCS CONC: _____mg/l

E.I. DuPont de Nemours 29082- 197 COC:

	I07931	I07931	I07931	I07931	I07931	I07931	I07931	I07931	I07931	I07931	I07931	I07931
SAMPLE #												
BOTTLE #												
INITIAL												
RESIDUAL												
DEPLETION												
SEED CORRECTION												
% OF SAMPLE												
BOD MG/L												

BOD CONC: _____ mg/l

EARTH TECH ENVIRONMENTAL LABORATORY-GR

Test #: 1151. 1- 409.51
Parameter: VOL'S IN DWTR BY 502.2
Method: VOLS/P&T/GC/WTR
Ref. cit.: USEPA-502.2 Unit: ug/l

Client: USEPA Performance Evaluation
Project: 29063 Water Supply Analysis
USEPA WS Study
Submittal: 8 WS Study-35
Sample: 105729 Volatiles #3
Proj Chem: DEK
Hold date: 23-DEC-1994 QC: LV2 COC:
Lab due date: 24-JAN-1995
Client due date: 25-JAN-1995 C=1 F=0 H=1 R=0
Received date: 12-DEC-1994

Benchsheet ID: 125868
Analyst: _____
Date Run: _____
Instrument #: _____
Stock Std: _____
Rack#: _____
Reviewed by: _____
QC Batch Number: _____

Initial wt./vol.: _____
Final volume: _____
% Solids: _____
Dilution factor: _____
Batch Number: _____
Batch Owner: _____
Batch Opened Date: _____
Batch Seq: _____

	Parameter	P	ODL	Result	LCL	UCL
42	1,2-DICHLOROBENZENE			< 1.0		
43	1,2-DIBROMO3CHLOROPROPANE			< 1.0		
44	1,2,4-TRICHLOROBENZENE			< 1.0		
45	HEXACHLOROBUTADIENE			< 1.0		
46	1,2,3-TRICHLOROBENZENE			< 1.0		
48	BENZENE			< 1.0		
49	TOLUENE			< 1.0		
50	ETHYLBENZENE			< 1.0		
51	XYLENE, META			< 1.0		
52	XYLENE, PARA			< 1.0		
53	XYLENE, ORTHO			< 1.0		
55	STYRENE			< 1.0		
56	ISOPROPYLBENZENE			< 1.0		
57	N-PROPYLBENZENE			< 1.0		
58	BROMOBENZENE			< 1.0		
59	1,3,5-TRIMETHYLBENZENE			< 1.0		
60	TERT-BUTYLBENZENE			< 1.0		
61	1,2,4-TRIMETHYLBENZENE			< 1.0		
62	SEC-BUTYLBENZENE			< 1.0		
63	PARA-ISOPROPYLTOLUENE			< 1.0		
64	N-BUTYLBENZENE			< 1.0		
65	NAPHTHALENE			< 1.0		
1	CL-2-FLB-(HALL)-SUR			_____%	47.55	145.71
2	CL-2-FLB-(PID)-SUR			_____%	81.06	171.18
3	4-CLT (HALL)-SUR			_____%	65.83	130.99
4	3-BCB (HALL)-SUR			_____%	69.91	131.11
5	FLUOROBENZENE (PID)-SUR			_____%	43	145
6	AAA-TFT (PID)-SUR			_____%	71.04	123.6
7	PCE (PID)-SUR			_____%	69.31	133.81
8	4-CLT (PID)-SUR			_____%	90.2	135.5
9	3-BCB (PID)-SUR			_____%	91	143.8

* - Result < corresponding ODL

EARTH TECH ENVIRONMENTAL LABORATORY-GR

Figure 22

10-JAN-95 Confirm: REG RC1 RC2 RMS

FRACTION BENCHSHEET
VOLATILE ORGANIC LAB

Test #: 1151. 1- 409.51
Parameter: VOL'S IN DWTR BY 502.2
Method: VOLS/P&T/GC/WTR
Ref. cit.: USEPA-502.2 Unit: ug/l

Client: USEPA Performance Evaluation
Project: 29063 Water Supply Analysis
USEPA WS Study
Submittal: 8 WS Study-35
Sample: 105729 Volatiles #3
Proj Chem: DEK
Hold date: 23-DEC-1994 QC: LV2 COC:
Lab due date: 24-JAN-1995
Client due date: 25-JAN-1995 C=1 F=0 H=1 R=0
Received date: 12-DEC-1994

Benchsheet ID: 125868
Analyst: _____
Date Run: _____
Instrument #: _____
Stock Std: _____
Rack#: _____
Reviewed by: _____
QC Batch Number: _____

Initial wt./vol.: _____
Final volume: _____
% Solids: _____
Dilution factor: _____
Batch Number: _____
Batch Owner: _____
Batch Opened Date: _____
Batch Seq: _____

	Parameter	P	ODL	Result	LCL	UCL
1	DICHLORODIFLUOROMETHANE			< 1.0		
2	CHLOROMETHANE			< 1.0		
3	VINYL CHLORIDE			< 1.0		
4	BROMOMETHANE			< 1.0		
5	CHLOROETHANE			< 1.0		
8	TRICHLOROFLUOROMETHANE			< 1.0		
9	1,1-DICHLOROETHYLENE			< 1.0		
10	METHYLENE CHLORIDE			< 1.0		
11	TRANS-1,2-DICHLOROETHYLEN			< 1.0		
12	1,1-DICHLOROETHANE			< 1.0		
13	2,2-DICHLOROPROPANE			< 1.0		
14	CIS-1,2-DICHLOROETHYLENE			< 1.0		
15	BROMOCHLOROMETHANE			< 1.0		
16	CHLOROFORM			< 1.0		
17	1,1,1-TRICHLOROETHANE			< 1.0		
18	1,1-DICHLOROPROPYLENE			< 1.0		
19	CARBON TETRACHLORIDE			< 1.0		
20	1,2-DICHLOROETHANE			< 1.0		
21	TRICHLOROETHYLENE			< 1.0		
22	1,2-DICHLOROPROPANE			< 1.0		
23	BROMODICHLOROMETHANE			< 1.0		
24	DIBROMOMETHANE			< 1.0		
26	CIS-1,3-DICHLOROPROPYLENE			< 1.0		
27	TRANS-1,3-DICHLOROPROPENE			< 1.0		
28	1,1,2-TRICHLOROETHANE			< 1.0		
29	1,3-DICHLOROPROPANE			< 1.0		
30	TETRACHLOROETHYLENE			< 1.0		
31	DIBROMOCHLOROMETHANE			< 1.0		
32	1,2-DIBROMOETHANE			< 1.0		
33	CHLOROBENZENE			< 1.0		
34	1,1,1,2-TETRACHLOROETHANE			< 1.0		
35	BROMOFORM			< 1.0		
36	1,1,2,2-TETRACHLOROETHANE			< 1.0		
37	1,2,3-TRICHLOROPROPANE			< 1.0		
38	2-CHLOROTOLUENE			< 1.0		
39	4-CHLOROTOLUENE			< 1.0		
40	1,3-DICHLOROBENZENE			< 1.0		
41	1,4-DICHLOROBENZENE			< 1.0		

* - Result < corresponding ODL

EARTH TECH ENVIRONMENTAL LABORATORY-GR

Fig. 23

10-JAN-95 INORGANIC BENCHSHEET — SPECTROPHOTOMETRIC ** QC ** PAGE 1

Test#: 399. 1- 259.01
Parameter: FLUORIDE
Method: FL/ION-ELEC/WTR ODL: 0.10
Ref. Cit.: USEPA-340.2 Unit: mg/l

Instrument #:
Benchsheet ID: I25866
Owner:
Batch Opened Date:
Supervisor:
Est anal hrs: .25
Act anal hrs:
Samples in batch: 1

Comments:

	Anl.	Run Date	Regressed Value	Dilution factor	mg/l	% Sol	Wt/dil factor	Reported Conc.	ODL	Spike Qty	% rec/dif	QH
BLK:										XXXXXXXX		
										XXXXXXXX		
LCS: Stk												
SPK1: Stk Smp												
SPK2: Stk Smp												
MSD: Smp		XXXXXXX	XXXXXXXXXX	XXXXXXX	XXXXX	XXX	XXXXXXXXXX	XXXXXXXXXX	XXXXXXX	XXXXXXX		
		XXXXXXX	XXXXXXXXXX	XXXXXXX	XXXXX	XXX	XXXXXXXXXX	XXXXXXXXXX	XXXXXXX	XXXXXXX		
DUP: Smp										XXXXXXX		
										XXXXXXX		
SPK1: Stk Smp												
SPK2: Stk Smp												
MSD: Smp		XXXXXXX	XXXXXXXXXX	XXXXXXX	XXXXX	XXX	XXXXXXXXXX	XXXXXXXXXX	XXXXXXX	XXXXXXX		
		XXXXXXX	XXXXXXXXXX	XXXXXXX	XXXXX	XXX	XXXXXXXXXX	XXXXXXXXXX	XXXXXXX	XXXXXXX		
DUP: Smp										XXXXXXX		
										XXXXXXX		
MPB: QCB#										XXXXXXX		
										XXXXXXX		
LFB: Stk QCB#												
CRDL: Stk												

QC Type	Range	Active Lcl	Active Ucl
DUP	1		22.04
DUP	2		12.97
LCS	1	81.02	120.74
MPB	1		10
MSD	1		35
MSD	2		25
SPK	1	65.69	128.75

EARTH TECH ENVIRONMENTAL LABORATORY-GR

Figure 24

Test#: 399. 1- 259.01
Parameter: FLUORIDE
Method: FL/ION-ELEC/WTR ODL: 0.10
Ref. Cit.: USEPA-340.2 Unit: mg/l

Batch: _____

	STD VAL	OBS VAL	WKG STD NUMBER
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			

Instrument #:
Benchsheet ID: I25866
Owner:
Batch Opened Date:
Supervisor:
Est anal hrs: .25
Act anal hrs:
Samples in batch: 1
Stock std #:
Wavelength (nm):
Cell path (mm):

Comments:

Client..... Submittal Location	Sample	COC	QC	Anl.	Run Date	Regressed Value	Dilution factor	mg/l	% Sol	Wt/dil factor	Reported Conc.	ODL	QH	Spike Qty	% rec/dif
ICV: Stk															
														XXXXXXXX	
ICB:														XXXXXXXX	
USEPA Performance Evaluation															
29063- 8	105720		LV2												
Nitrate Ni															
CCV: Stk		Rep													
														XXXXXXXX	
CCB:		Rep												XXXXXXXX	

Figure 25

8-16-94 KLV 8:00am

7163 DM 8/16

dm 8-29

12-AUG-94 INORGANIC BENCHSHEET -- SPECTROPHOTOMETRIC PAGE 1

Test#: 408. I- 280.01
Parameter: PHOSPHORUS, ORTHO
Method: OPHOS/COLOR/WTR ODL: 0.01
Ref. Cit.: USEPA-365.2 Unit: mg/l

	STD VAL	OBS VAL	WKG STD NUMBER
1.	0.01	0.010	167K.1.1.1.3.1
2.	0.025	0.027	167K.1.1.1.2.1
3.	0.25	0.300	167K.1.1.1.2
4.	0.5	0.609	167K.1.1.1.1
5.	1.0	1.211	167K.1.1.1.3
6.			
7.			
8.			

Instrument #:
Benchsheet ID: II2638
Owner: GCH
Date run: 8-12-94
Supervisor: BJD
Est anal hrs: .75
Act anal hrs:
Samples in batch: 3

Comments:

Stock std #: 167K.1.1
Wavelength (nm): 880
Cell path (mm): 20

Client Submittal Location	Sample	COC	QC	Regressed Value	Wt/dil Factor	Reported Conc.	ODL	Spike Qty.	% rec/dif
ICB:						<0.01		XXXXXXXX XXXXXXXX	0
ICV: Stk 167K.1.1.1.2.1						0.025		0.025	100
Paulstra-CRC 32466- 1 MW-4	94789 *		LV1			<0.01			
Paulstra-CRC 32466- 1 MW-6	94790 *		LV1			<0.01			
Paulstra-CRC 32466- 1 MW-10	94791 *		LV1			<0.01			

ANALYSIS NARRATIVE
SAMPLE NO: 94789, 94790, 94791
Sample was filtered prior to ortho phosphorus analysis.

CCB:						<0.01		XXXXXXXX XXXXXXXX	0
CCV: Stk 167K.1.1.1.1						0.49		0.5	98

EARTH TECH ENVIRONMENTAL LABORATORY-GR

Figure 26

12-AUG-94 INORGANIC BENCHSHEET -- SPECTROPHOTEMETRIC ** QC ** PAGE 1

TestN: 408. 1- 280.01
Parameter: PHOSPHORUS, ORTHO
Method: ORTHOS/COLOR/WTR MDL: 0.01
Ref. Cit.: USEPA-365.2 Unit: mg/l

Instrument #:
Benchsheet ID: II2668
Owner: GEH
Date run: 8-12-94
Supervisor:
Est anal hrs: .75
Act anal hrs:
Samples in batch: 3

Comments:

Stock std #:
Wavelength (nm):
Cell path (mm):

			Regressed Value	Wt/dil factor	Reported Conc.	MDL	Spike Qty	% rec/dif	
MPB:					<0.01		XXXXXXXX XXXXXXXX	0	✓
LCS:	Stk	WW# 220 A			0.40		0.38	105	✓
SPK:	Stk	167K.1.1 Smp	94791		0.53		0.5	106	✓
MSD:	Stk		Smp						
DUP:		Smp	94791		<0.01		XXXXXXXX XXXXXXXX	0	✓
SPK:	Stk		Smp						
MSD:	Stk		Smp						
DUP:		Smp					XXXXXXXX XXXXXXXX		
MPB:							XXXXXXXX		
LFS:	Stk								

QC Typ	Range	Active Lcl	Active Ucl
DUP	1		20.88
DUP	2		7.93
LCS	1	89.16	112.14
MPB	1		10
MSD	1		35
MSD	2		23
SPK	1	88.02	117.42

EARTH TECH ENVIRONMENTAL LABORATORY-GR

Figure 26

INORGANIC BENCHSHEET - [GRAVIMETRIC]

16- Dec - - File Name : '121694rd.ww1'

Test Number : 9. 1 - 10. 02
Paramater : Residue, Disolved
Matrix : WW
Ref. Cit. : USEPA-160.1

Cycle 1 Temp : Start 180 °C End 180 °C
Cycle 2 Temp : Start 180 °C End 180 °C
Unit : mg/l
ODL : 1

Owner : GEH
Date Run : 12-16-94
Supervisor : BJD

Client	Sample	RepConc	Dish	WtDishRes1	WtDishRes2	WtDish	SampAmt	ODL
KENT CO.	106283	753	10	131.4461	131.4456	131.3708	100.0000	1
KENT CO.	106284	377	11	123.4886	123.4882	123.4509	100.0000	1
KENT CO.	106285	377	12	130.3341	130.3336	130.2964	100.0000	1
KENT CO.	106286	780	13	120.4236	120.4232	120.3456	100.0000	1
KENT CO.	106287	594	14	120.5840	120.5835	120.5543	50.0000	1

QC	Stk	Sample	RepConc	SpkQty	Dish	WtDishRes1	WtDishRes2	WtDish	SampAmt	ODL	%RecDif
BLK		0	< 1	0.0	999	117.9183	117.9183	117.9183	100.0000	1	0.00
LCS	WW#291	0	198	200.1	700	122.8662	122.8662	122.8464	100.0000	1	98.95
SPK		0		0.0	800	0.0000	0.0000	0.0000	0.0000	1	0.00
DUP		106287	582	0.0	15	119.7324	119.7323	119.7033	50.0000	1	2.04
SPK	264G.2	106287	1240	659.2	16	123.2377	123.2378	123.1759	50.0000	1	98.00
DUP		0		0.0	901	0.0000	0.0000	0.0000	0.0000	1	0.00
DUP		0		0.0	902	0.0000	0.0000	0.0000	0.0000	1	0.00
DUP		0		0.0	903	0.0000	0.0000	0.0000	0.0000	1	0.00
DUP		0		0.0	904	0.0000	0.0000	0.0000	0.0000	1	0.00

Figure 27

4.1.2.5 Management Reports

Several reports are provided within the TriMatrix laboratory system to help monitor operational conditions of the laboratory. These reports are provided or compiled both electronically and for various activities including but not limited to: jobs due report, workload-loading reports, sales summaries, client/project/submittal informational reports, etc.

The flow of information from these various reports is geared to a variety of personnel within the management structure of the laboratory and to some persons outside the laboratory. Information is generally provided to employees outside the laboratory for corporate management decisions or in providing information to a particular client about their project.

Examples of a few management type reports are presented in Figures 28 to 32.

01-MAR-19[redacted]

EARTH TECH ENVIR[redacted]NTAL LABORATORY-GR
OFFICE JOB[redacted]E REPORT

PA[redacted]1

Projec[redacted]ype: CCS

CLIENT DUE DATE	PROJ. CHEM.	SUBMITTAL		CLIENT	PROJECT	FIRST SAMPLE	NUM.
02-MAR-1995	GJH	30854-38	6	EARTH TECH Laboratories -Livon	Analysis Of Wastewater	111303	4
02-MAR-1995	GJH	32420-83	6	EARTH TECH Laboratories -Livon	Wastewater Analysis	111088	6
06-MAR-1995	GJH	30822-17	3	EARTH TECH Laboratories -Livon	TCLP Analysis	111560	1
06-MAR-1995	GJH	32630-4	3	EARTH TECH Laboratories -Livon	MONSANTO	111040	2
07-MAR-1995	GJH	30854-37	3	EARTH TECH Laboratories -Livon	Analysis Of Wastewater	111307	2
07-MAR-1995	GJH	30854-39	3	EARTH TECH Laboratories -Livon	Analysis Of Wastewater	111296	7
07-MAR-1995	GJH	31573-234	3	EARTH TECH Laboratories -Livon	Mercury Analysis	111561	3
08-MAR-1995	GJH	32403-38	3	EARTH TECH Laboratories -Livon	Water Analysis	111591	2
08-MAR-1995	GJH	32266-37	3	White Construction, Inc.	Eli Lilly and Company	111422	27
09-MAR-1995	GJH	32420-84	3	EARTH TECH Laboratories -Livon	Wastewater Analysis	111564	6

Figure 28

2 - Partially Received; 3 - Completely Received; 4 - Compiled; 5 - Approved 6 - Released

Class ID	Prev Weeks	Sunday 01-JAN-95	Monday 02-JAN-95	Tuesday 03-JAN-95	Wednesday 04-JAN-95	Thursday 05-JAN-95	Friday 06-JAN-95	Saturday 07-JAN-95	Weekly Total	Max Per Week	Percent
ASVO	31	0	3	4	12	4	2	0	25	100	25%
QCV 8240	1	0	0	0	0	0	0	0	0	100	0%
QCV 824	2	0	0	0	0	0	0	0	0	100	0%
QCV TCLP	7	0	0	0	0	0	0	0	0	40	0%
GC-AIRWORK	0	0	0	0	0	0	0	0	0	20	0%
GCVO	17	0	0	0	0	0	0	0	0	240	0%

Figure 29

[redacted] ANALYSIS WITH A LAB DUE DATE) OR = 01-Mar-1994 AND [redacted] = 01-Mar-1995*

[redacted]83	ADRIAN MDNR 3.		USEPA-8121	WTR	1						
109284	ADRIAN MDNR 3.		USEPA-8121	WTR	2						

[redacted] TURNAROUND DAYS: 21 PROJECT CHEMIST: NBK QC TYPE: LV1 COC: E
32640-3 FORECAST AMT: $427.50 LAB DUE DATE: 20-FEB-1995 (MON)
General Electric-Holland February 9,1995 Submittal

110528	VOLS BY 8021/SCAN 1+2 LST		USEPA-8021	TCLP	2						
110529	VOLS BY 8021/SCAN 1+2 LST		USEPA-8021	TCLP	2						

[redacted] TURNAROUND DAYS: 21 PROJECT CHEMIST: GKR QC TYPE: LV1 COC: E
29011-230 FORECAST AMT: $65.00 LAB DUE DATE: 27-FEB-1995 (MON)
Outfall Monitoring Northkent Landfill February 1995 Week #2

110440	OXYGEN, % SATURATION		USEPA-360.2	WW	0	OXYGEN, DISSOLVED (FIELD)		USEPA-360.2	WW	0	
	PH (FIELD)		USEPA-150.1	WW	0	TEMPERATURE, CELSIUS		USEPA-170.1	WW	0	
110441	OXYGEN, % SATURATION		USEPA-360.2	WW	0	OXYGEN, DISSOLVED (FIELD)		USEPA-360.2	WW	0	
	PH (FIELD)		USEPA-150.1	WW	0	TEMPERATURE, CELSIUS		USEPA-170.1	WW	0	

[redacted] Paul, Minnesota TURNAROUND DAYS: 21 PROJECT CHEMIST: RWH QC TYPE: LV2 COC: E
31665-22 FORECAST AMT: $2,120.00 LAB DUE DATE: 28-FEB-1995 (TUE)
Cordova Priority Pollutan 3M Request # N1262

110391	3M CORDOVA PEST/PP		USEPA-608	WW	2						

[redacted] TURNAROUND DAYS: 14 PROJECT CHEMIST: GKR QC TYPE: LV1 COC: E
31667-4 FORECAST AMT: $668.00 LAB DUE DATE: 28-FEB-1995 (TUE)
Sludge Analysis 15-February-1995 Sampling

110863	PCB'S USEPA-8080 SCAN		USEPA-8080	SLG	2						

[redacted] TURNAROUND DAYS: 14 PROJECT CHEMIST: GLW QC TYPE: LV3 COC: E
32349-3 FORECAST AMT: $425.00 LAB DUE DATE: 28-FEB-1995 (TUE)
Raw Materials Analysis Perrigo 04366

110874	METHANOL		USEPA-8015	WASTE	0						
110876	ETHANOL		USEPA-8015	WASTE	0	ISOPROPANOL		USEPA-8015	WASTE	0	

[redacted] TURNAROUND DAYS: 14 PROJECT CHEMIST: CJS QC TYPE: LV1 COC: E
32663-1 FORECAST AMT: $888.00 LAB DUE DATE: 28-FEB-1995 (TUE)
Wastewater for Volatiles and Lead February 16,1995 sampling

110997	TROY ALCOHOLS		USEPA-8015	WW	0						

Figure 30

SUBMITTAL NOT MEETING CLIENT DUE DATE

CLIENT	SUBMITTAL	COMPILED DATE	APPROVED DATE	RELEASED DATE	MAILED DATE	CLIENT DUE DATE	LAB DUE DATE	TURN AROUND	PROJ CHEM
[redacted]	29015-242	02/14/95	02/20/95	02/20/95		02/24/95	02/21/95	21	RWH
Last Approved Date	Lab Id								
[redacted]	29491-168	02/13/95	02/20/95	02/20/95		02/24/95	02/21/95	21	RWH
Last Approved Date	Lab Id								
[redacted]	32641-1	02/23/95				02/24/95	02/21/95	21	GLW
Last Approved Date	Lab Id								
[redacted] - TN	30436-488	02/23/95	02/24/95	02/24/95		02/24/95	02/22/95	9	GLW
Last Approved Date	Lab Id								
[redacted]	32275-10	02/22/95				02/24/95	02/22/95	21	GLW
Last Approved Date	Lab Id								
[redacted]	29928-164	02/23/95				02/24/95	02/23/95	21	RVB
Last Approved Date	Lab Id								

Figure 31

FOR SAMPLES WITH A SAMPLE LOG DATE FROM 26-Feb-1994 TO 26-Feb-1996

CLIEN	SUBMITTAL	P	Ch	SALES AMT	SALES + ADD	PROJECT NUM		DUE DATE	SAMPLE			S	TYPE
	16-December-1994 soils		GKR	10,860.00	10,860.00	32454-	3	09-JAN-95	106695	15	5		CCS
	Jan 12 Arrival MR		RVB	128.00	128.00	32583-	37	18-JAN-95	108488	4	6		CCS
	Jan 16 Arrival GM		RVB	32.00	32.00	32583-	38	20-JAN-95	108593	1	6		CCS
	Jan 16 Arrival NN		RVB	12.00	12.00	32583-	39	20-JAN-95	108594	1	6		CCS
	Jan 18 Arrival PL		RVB	480.00	480.00	32583-	42	24-JAN-95	108872	40	6		CCS
	Jan 19 Arrival MR		RVB	444.00	444.00	32583-	43	25-JAN-95	108771	17	6		CCS
	Jan 24 Arrival MR		RVB	152.00	152.00	32583-	44	30-JAN-95	109233	6	6		CCS
	Jan 24 Arrival NN		RVB	80.00	80.00	32583-	45	30-JAN-95	109240	5	6		CCS
	Jan 30 Arrival MR		RVB	340.00	340.00	32583-	46	03-FEB-95	109617	25	6		CCS
	January 20,1995 Submittal		GLW	17,800.00	17,800.00	31447-	4	07-FEB-95	109061	40	6		CCS
	Feb 2 Arrival MR		RVB	36.00	36.00	32583-	47	08-FEB-95	109835	3	6		CCS
	Feb 2 Arrival NN		RVB	96.00	96.00	32583-	48	08-FEB-95	109838	8	6		CCS
	Jan 1995 Samples		RVB	29,775.00	29,775.00	29699-	23	10-FEB-95	109351	50	3		CCS
	Feb 7 Arrival MR		RVB	648.00	648.00	32583-	49	13-FEB-95	110194	29	6		CCS
	Feb 7 Arrival NN		RVB	288.00	288.00	32583-	50	13-FEB-95	110221	19	6		CCS
	January 27, 1995 Submitta		GKR	12,396.00	12,396.00	32454-	9	15-FEB-95	109552	18	6		CCS
	Jan. 27, 1995 - Soils		GKR	6,094.00	6,094.00	32454-	10	15-FEB-95	109545	7	6		CCS
	TN 15216		GLW	300.00	300.00	31834-	24	19-FEB-95	110769	6	4		CCS
	February 9,1995 Submittal		NBK	710.12	710.12	32640-	3	20-FEB-95	110528	2	3		CCS
	Feb 14 Arrival NN		RVB	60.00	60.00	32583-	51	20-FEB-95	110571	5	6		CCS
	Feb 14 Arrival MR		RVB	560.00	560.00	32583-	52	20-FEB-95	110576	35	6		CCS
	February, 1995 Samples		GLW	4,211.00	4,211.00	32641-	1	21-FEB-95	110036	28	4		CCS
	TN 15198,199,203		GLW	372.00	372.00	30436-	488	22-FEB-95	110827	11	6		CCS
	February 6,1995 Submittal		NBK	5,814.00	5,814.00	32509-	13	22-FEB-95	110129	32	4		CCS
	February 4, 1995 Samples		RVB	898.00	898.00	29928-	164	23-FEB-95	110191	2	4		CCS
	TN 15268		GLW	52.50	52.50	30436-	492	24-FEB-95	111119	1	3		CCS
	February 8,1995 Submittal		NBK	1,800.00	1,800.00	32509-	14	24-FEB-95	110298	10	4		CCS
	03-February-1995 Sampling		GKR	1,408.00	1,408.00	32454-	11	24-FEB-95	110320	3	3		CCS
	Specila Coliform Test		GJH	75.00	160.00	32666-	1	26-FEB-95	111100	5	3		CCS
	02-February-1995 Sampling		GLW	216.00	216.00	30272-	128	27-FEB-95	110123	2	3		CCS
	TN 15218, 15217		GLW	654.00	654.00	30436-	487	27-FEB-95	110815	19	6		CCS
	TN 15221,234,241,242,243		GLW	405.50	405.50	30436-	490	27-FEB-95	110993	7	3		CCS
	TN 15223,244,222,240		GLW	346.50	346.50	30436-	491	27-FEB-95	111072	9	3		CCS
	19-January-1995 Sampling		GKR	60.00	60.00	29642-	17	27-FEB-95	111097	3	3		CCS
	February 1995 Purge Wells		RVB	845.00	845.00	29158-	138	27-FEB-95	110431	9	3		CCS
	February 9, 1995 Samples		GLW	736.00	736.00	31249-	140	27-FEB-95	110687	46	3		CCS
	Feb 14 Samples		RVB	4,550.00	4,550.00	29140-	11	27-FEB-95	110654	7	3		CCS
	TN 15255,260,257,256		GLW	598.50	598.50	30436-	493	28-FEB-95	111120	19	3		CCS
	February 10,1995 Submittal		NBK	360.00	360.00	32509-	15	28-FEB-95	110520	2	4		CCS
	15-February-1995 Sampling		GKR	668.00	668.00	31667-	4	28-FEB-95	110863	1	3		CCS
	15-February-1995 sampling		GKR	172.00	172.00	31777-	71	28-FEB-95	110860	2	4		CCS
	13-February-1995 Sampling		GKR	28.80	28.80	29280-	28	28-FEB-95	110858	1	3		CCS
	15-February-1995 Sampling		GKR	694.00	694.00	32471-	2	28-FEB-95	110856	2	3		CCS
	February 22, 1995 Sample		RVB	85.00	85.00	29841-	194	28-FEB-95	111353	1	3		CCS
	Feb 9 Samples		RVB	898.00	898.00	29928-	165	28-FEB-95	110471	2	3		CCS
	February 9, 1995 Samples		GLW	270.00	270.00	30505-	10	28-FEB-95	110869	2	6		CCS
	TN 15269		GLW	190.50	190.50	30436-	494	01-MAR-95	111140	3	3		CCS
	EARTH TECH Livonia:114907		GJH	465.00	465.00	30704-	145	01-MAR-95	111094	3	4		CCS

Figure 32

4.1.2.6 Quality Assurance Reports

Quality assurance reports play a vital role in the management of the quality system. Quality systems must be closely scrutinized in order to monitor, maintain, adjust and add procedures or systems to meet existing and new QA objectives of the laboratory.

Several quality assurance reports are created in this monitoring maintenance effort. These reports serve different functions and are designed to inform the ultimate user. In the case of a client/invoice report, the quality assurance data is presented to facilitate the objectives of the project requirements from data assessment through full 3rd party data validation.

Internal quality assurance reports are created through both the TriMatrix LIMS system and external means. Some of the QA reports available through our LIMS includes:

Result Lists/Shewart Charts
Biased Statistics
Control Limit Comparison Report
Statistic Summary Report
Analytical Batch Detail Report
QC Batch Detail Report
Deviant Statistics
Locked Statistics
Analyst Certification
LIMS Qualifiers

Discussions and examples of these and other quality assurance reports are presented in section 4.1.2.9-F and section 4.9.

4.1.2.7 Client Invoice/Quality Control Reporting

Client/Invoice Reports are produced both automatically upon the completion of a submittal or sample delivery group. The initial report produced or "preliminary report" is an exact line printer replicate of the final report.

Preliminary reports are sorted, placed into the original submittal or project folder that was created at log-in and forwarded to the appropriate project chemist.

The project chemist reviews the preliminary report to validate that the data generated has fulfilled specifications originally created or requested for this project. The project chemist also validates the existence of the correct qualifications for when specifications have not been fulfilled.

Upon review, the project chemist approves the preliminary report, thus making it available for the final report generation process. The printing of the final report is performed by the laboratory data coordinator. All final reports are forwarded to the project chemist for final signature and release. Each signed report is forwarded to the laboratory secretary for mailing to the client and the laboratory project files. Dates are recorded as each submittal or report is mailed. Quality assurance data for a submittal or delivery package is presented as part of the both the preliminary and final reports. The extent of the quality assurance data presented as well as a description of part of each reporting level of the final report is present in subsections A-E.

A) Level 1 - General Reporting Format

The ***Level 1*** reporting format is the general reporting format and is typically utilized for most laboratory projects. The ***Level 1*** package will consist of the following :

Report Description	***No. of Copies Mailed***
Cover Letter w/Project Chemist sign-off	1
Results of Analysis	2
Statement of Data Qualifications	2
Invoice	1

Report/Invoice Options:

Results of Analysis	***Invoice***
a) General Format (Landscape or Portrait)	a) Internal Client
b) CAS Report	b) External Client
c) Detection Limit (fractions only)	c) Itemized[1]
	d) Lump Sum Invoice[2]

Notes:

1. The itemized invoice will list all samples and analyses on an individual basis.
2. The Lump Sum Invoice will print only single line items on the invoice with the corresponding amount. (example: "Program A Testing" $150.00).

B) Level 2 - Batch QC Reporting Format

The ***Level 2*** reporting format will consist of items from the ***Level 1*** reporting package, with the exception of batch method QC. This format will include the following:

Report Description	***No. of Copies Mailed***
Cover Letter w/Project Chemist sign-off	1
Results of Analysis	2
Methods Page	2
Batch QA/QC Results MPBs, LFBs, BLKs, LCSs & SURs	2
Statement of Data Qualifications	2
Field Chain-of Custody Form (COC)	1
Invoice	1

Report/Invoice Options:

Results of Analysis	***Invoice***
a) General Format (Landscape or Portrait)	a) Internal Client
b) CAS Report	b) External Client
c) Detection Limit (fractions only)	c) Itemized
	d) Lump Sum Invoice

C) Level 3 - Data Assessment Reporting Format

The ***Level 3*** reporting format will consist of all items from the ***Level 2*** package with the addition client specific matrix QC and the Analysis/Pretreatment Date Summary report. This format will include the following:

Report Description	***No. of Copies Mailed***
Cover Letter w/Project Chemist sign-off	1
Results of Analysis	2
Methods Page	2
Batch QA/QC Results (MPBs, LFBs, BLKs, LCSs & SURs	2
Client Specific Matrix QC (SPKs, MSDs and/or DUPs)	2
Pretreatment/Date Summary Page*	2
Statement of Data Qualifications	2
Field Chain-of Custody Form (COC)	1
Invoice	1

*The "Analysis/Pretreatment Date Summary Page" will present each sample and all tests with the dates of digestion, extraction and analysis and their corresponding expiration dates. This report will allow the data reviewer to quickly check for analysis hold time failures.

Report/Invoice Options:

Results of Analysis	***Invoice***
a) General Format (Landscape or Portrait)	a) Internal Client
b) CAS Report	b) External Client
c) Detection Limit (fractions only)	c) Itemized
	d) Lump Sum Invoice

D) Level 4 - Data Validation Reporting Format

The *Level 4* reporting format will consist of all items presented in the *Level 3* reporting package with the addition of providing raw data for all analyses. This format has been broken down into lab areas and will include the following:

General Reporting:

Report Description	*No. of Copies Mailed*
Cover Letter w/Project Chemist sign-off	1
Results of Analysis	2
Methods Page	2
Batch QA/QC Results (MPBs, LFBs, BLKs, LCSs, SURs)	2
Client Specific Matrix QC (SPKs, MSDs and/or DUPs)	2
Pretreatment/Date Summary Page	2
Statement of Data Qualifications	2
Internal Chain-of-Custody Forms (if requested)	1
Field Chain-of Custody Form (COC)	1
pH integrity Log forms from Log-in	1
Invoice	1
Batch Detail Reports for all batches	1

Report/Invoice Options:

Results of Analysis	*Invoice*
a) General Format (Landscape or Portrait)	a) Internal Client
b) CAS Report	b) External Client
c) Detection Limit (fractions only)	c) Itemized
	d) Lump Sum Invoice

Level 4 - Data Validation Reporting Format

Area Specific Reporting (raw data packages)

Client will be allowed to pick and choose from any of the items below.

GC/MS Reportables

*Tuning data for BFB and/or DFTPP

*Sample chromatograms and quantitation reports including all QC samples

*Sample chromatograms and quantitation reports for Initial and Continuing Calibrations

*Mass spectra, including reference spectra for all positive results

*Initial calibration response factor summaries with %RSD

*Continuing calibration response factor summaries with %RSD

*Library Search/Semi-Quantitation of TICs (if requested)

*Internal Standard Area and Retention Time Summary Report

GC Reportables

*Sample chromatograms and quantitation reports including all QC samples

*Record of standard curves

*Initial calibration response factor summaries with %RSD

*Continuing calibration response factor summaries with %RD

Retention Time Window Summary Table

*Second column confirmation and quantitation (if applicable)

*GC/MS confirmation (if applicable)

Metals Reportables

*Instrument printouts for all samples, standards and QC samples.

*preparation logs/forms for samples, QC samples and blanks

*Initial and continuing calibration verifications

*ICP inter element correction factors

*ICP linear range table

*ICP Serial Dilution's

Level 4 - Data Validation Reporting Format

Area Specific Reporting (raw data packages)

Inorganic/Nonmetals Reportables

*Instrument printouts for all samples, standards and QC samples.(if applicable)

*preparation logs/forms for samples, QC samples and blanks

*Initial and continuing calibration verifications

*Copies of all bench sheets used for reporting

Extraction Laboratory Reportables

*Extraction Summary forms for samples, QC samples and blanks

E) ***Level 5 - Contract Laboratory Program (CLP) Reporting Format***

The ***Level 5*** reporting package will consist of a CLP type report, presented as specified in the following USEPA Contract Laboratory Program statement of work outlines (SOW's):

Volatile Organics	(Aqueous/Solids) - CLP SOW 2/88
Semi-Volatile Organics	(Aqueous/Solids) - CLP SOW 2/88
Pesticides/PCB's	(Aqueous/Solids) - CLP SOW 2/88
Metals(except Mercury)	(Aqueous/Solids) - CLP SOW 10/91
Mercury	(Aqueous/Solids) - CLP SOW 2/88

General Reporting:

Report Description	***No. of Copies Mailed***
Cover Letter w/Project Chemist sign-off	1
Case Narrative	2
Methods Page	2
Pretreatment/Date Summary Page	2
Statement of Data Qualifications	2
Internal Chain-of-Custody Forms	1
Field Chain-of Custody Form (COC)	1
pH integrity Log forms from Log-in	1
Cooler temperature forms from Log-in	1
Invoice	1

Report/Invoice Options:

Results of Analysis	***Invoice***
a) CLP Format (Portrait)	a) Internal Client
	b) External Client
	c) Itemized
	d) Lump Sum Invoice

Level 5 - CLP Reporting Format

Area Specific Reporting (data packages)

GC/MS Reportables

*Form 1 - Sample Results

*Form 2 - Surrogate Results Report (SUR)

*Form 3 - Quality Control Data Report (SPK, MSD and LCS/LFB)

*Form 4 - Blank Summary Report (MPB and/or BLK)

*Form 5 - Tuning data for BFB and/or DFTPP

*Form 6 - Initial Calibration Report

*Form 7 - Continuing Calibration Report

*Form 8 - Internal Standard Area and Retention Time Summary Report (INS)

*Sample chromatograms and quantitation reports for all data

*Mass spectra, including reference spectra for all positive results

*Library Search/Semi-Quantitation of TICs (as requested)

GC Reportables

*Form 1 - Sample Results

*Form 2 - Surrogate Results Report (SUR)

*Form 3 - Quality Control Data Report (SPK, MSD and LCS/LFB)

*Form 4 - Blank Summary Report (MPB and/or BLK)

*Form 6 - Initial Calibration Report

*Form 7 - Calibration Verification Report

*Form 8 - Pesticide Analytical Sequence Report

*Form 9 - Pesticide Florisil Check

*Form 10 - Pesticide Identification Summary Report

*Sample chromatograms and quantitation reports including all QC samples

*Retention Time Window Summary Table

*Second column-confirmation and quantitation (if applicable)

*GC/MS confirmation (if applicable)

Level 5 - CLP Reporting Format

Area Specific Reporting (data packages)

Metals Reportables

*Form 1 - Sample Data

*Form 2A - Initial and Continuing Calibration Data Report (ICV and CCV)

*Form 2B - CRDL Standards Report (CRL)

*Form 3 - Blanks Report (ICB, CCB, BLK and/or MPB)

*Form 4 - ICP Interference Check Sample Report (IEC)

*Form 5A - Matrix Spike Recovery Report (SPK)

*Form 5B - Post Digestion Spike Report (PDS)

*Form 6 - Matrix Spike Duplicate Report (MSD)

*Form 7 - Laboratory Control Sample Report (LCS and/or LFB)

*Form 8 - Standard Additions Report (If required)

*Form 9 - ICP Serial Dilution's Report (If required)

*Form 10 - Instrument Detection Limits Report

*Form 11 - ICP Inter-Element Correction Factors Report

*Form 12 - ICP Linear Range Table

*Form 13 - preparation logs/forms for samples, QC samples and blanks

*Form 14 - Analysis Run Log (If Required)

*Instrument Raw Data printouts for all samples, standards and QC samples.

Inorganic/Nonmetals Reportables

*Instrument printouts for all samples, standards and QC samples.(if applicable)

*preparation logs/forms for samples, QC samples and blanks

*Initial and continuing calibration verifications

*Copies of all bench sheets used for reporting

Extraction Laboratory Reportables

*Extraction Summary forms for samples, QC samples and blanks

TABLE 2
Quality Assurance/Quality Control Type Designations
LIMS Abbreviations:

Method QC:

MPB	Method Preparation Blank
BLK	Daily Blank, Analytical/Instrument
LFB	Laboratory Fortified Blank
LCS	Laboratory Control Sample
IEC	ICP Interference Check Sample
CRL	Contract Required Detection Limit Standard
ICB	Initial Calibration Blank
ICV	Initial Calibration Verification
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification

Matrix QC:

SPK	Sample Matrix Spike
MSD	Sample Matrix Spike Duplicate
DUP	Sample Matrix Duplicate
PDS	Post Digestion Spike

Miscellaneous

SUR	Surrogate Spike
TIC	Tentatively Identified Compound
BFB	4-Bromofluorobenzene
DFTPP	Decafluorotriphenylphosphine
INS	Internal Standard

If a laboratory project is designated as a level 4 or higher, additional materials such as instrument raw data is also assembled as part of the final report. A flow chart illustrating the report generation process is presented in Figure 33.

TriMatrix Laboratories, Inc.

Report Generation Process

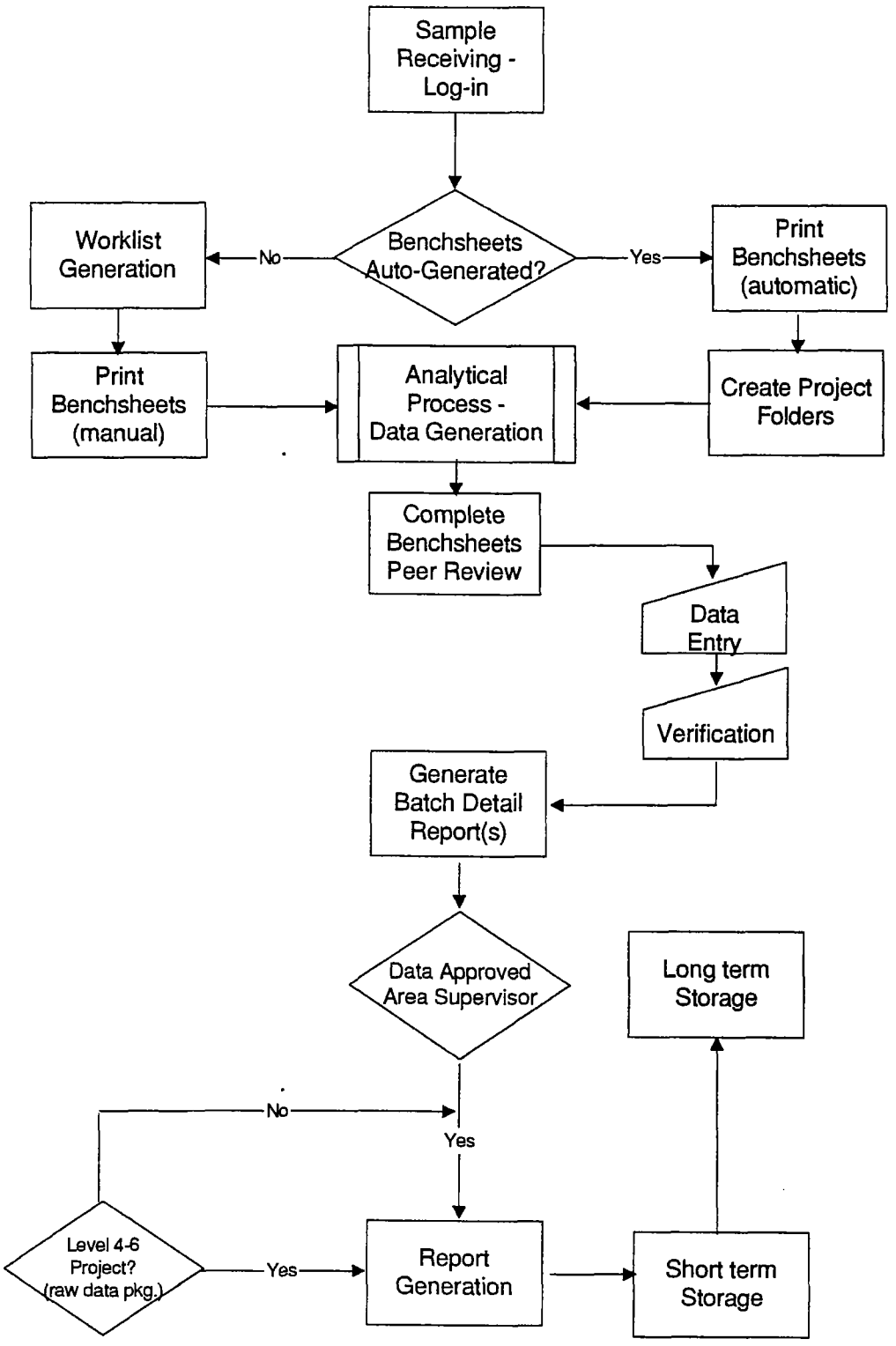


Figure 33

4.1.2.8 Project Files

The project file is the comprehensive record of every project completed at the TriMatrix laboratories. Each project file consists of a file folder set-up by the laboratory secretary, at the time of log-in. All project files are stored in a secure/strict limited-access area of the laboratory. The laboratory secretary is responsible for including the following in the project file:

- Initial project report/analysis plan/proposal
- All correspondence or documents mailed or received with the samples.
- All sample receiving/log-in forms.
- Chain-of-custody forms.
- Laboratory worksheets.
- Preliminary reports.
- Copy of the invoice.
- Final laboratory report including all reported quality control data.

All project files are stored on-site for 1 year and then off-site at a secured limited access storage facility for an additional 6 years.

4.1.2.9 Quality Control Documents

A) Analyst Notebooks

The lab notebooks are the daily records of all activities of an analyst, or group of analysts, working in the lab. The notebooks are bound and paginated. The notebook is cleanly labeled on the inside cover with the date issued, the analyst's name, and the date completed. There are several specific rules which are followed:

- All entries are in ink
- There are no erasures, obliteration's, or white outs allowed
- Corrections are single lined and initialed
- A new page is started each day or with every batch of samples
- Empty space is covered with a Z and signed and dated across the obtuse line
- Any and all work, observations, and errors are noted
- Problem areas identified
- Each page is signed and dated by the analyst performing the work.

When the instrument has just been repaired, a lamp changed, new column installed, detector repaired, or changed in any other manner, the log will also contain:

- A comment relative to the change or repair
- Reference page number to the Instrument Maintenance Log

The organic log books also contains the following information relative to GC and GCMS oven and column conditions UNLESS they are exactly as specified in the referenced method (which then will be commented on as such):

- column used (packing, diameter, length, type) o capillary as split or splitless
- current type and flow
- make-up flow if appropriate
- oven temperature and program if appropriate
- injector temperature
- detector temperature
- ion chamber voltage

B) Instrument Logbooks

Two different instrument logbooks are maintained on each laboratory instrument-an instrument run-log and an instrument maintenance log. Each log plays an important role in the documentation of daily instrument activities.

The instrument run-log is a bound and paginated log which is used to document all analytical determinations of a designated instrument. These determinations include not only sample analyses, but recordings of all calibration and calibration runs, quality control analyses and where applicable any instrument tuning activities.

The instrument run log also provides a chronology of each days analyses. This chronology plays an important role in the data validation process. All run logs are identified by instrument manufacturer name and model number, instrument serial number, and the starting and ending dates encompassed. All completed run logs are issued document control numbers, inventoried and properly archived.

The instrument maintenance log is a bound and paginated log which is used to track potential maintenance problems. The maintenance log is used to document whenever an instrument maintenance procedure, repair or modification activity is performed. All activities are documented by recording what was done, by whom and why. Other activities that are recorded in this log include routine daily functions such as septum changes on a GC injector or the cleaning of optical windows on an atomic absorption unit.

All completed maintenance logs are identified by instrument manufacturer name and model number, instrument serial number and the starting and ending dates encompassed. All maintenance logs are issued document control numbers, inventoried and properly archived.

C) Control Temperature Units

Each oven, incubator and all cold storage devices have their temperatures monitored and recorded on a daily basis. Within each controlled temperature unit is a thermometer which has been checked on a periodic basis against a NIST traceable thermometer. The schedule for these certifications is annually for mercury-in-glass and quarterly for all dial type thermometers.

All temperature readings and thermometer calibrations are recorded in a controlled temperature logbook. This log contains a page for each unit with detailed information on unit identification, serial number, laboratory location and designated operating temperature. Accompanying each unique log unit record page is a step by step corrective action procedure to be referenced when a temperature unit has fallen outside its predetermined operating range. An example of a controlled temperature log form and its corresponding corrective action procedure is presented in figures 34 and 35.

TriMatrix Laboratories, Inc.

Controlled Temperature Unit #5 (CTU5)
Daily Log Sheet

Unit Serial #	25AT-7	**Unit Description:**	BOD Refrigerator/Incubator
Thermometer #	Not Available	**Model Number**	Model 182
Correction Factor	< 0.1 C	**Location**	Organic Glassware Preparation Room
Control Windows	Low: 19 C High: 21 C	**Purpose**	BOD Incubation

Date	Initials	Time of First Reading	First Reading (C)	Time of Second Reading	Second Reading (C)	Adjustments/Observations/Comments

Figure 34

STANDARD OPERATING PROCEDURE
CORRECTIVE ACTION
"CONTROLLED TEMPERATURE UNITS"

Device: Randell Model 1020 Refrigerator

Operating Range: 1-5°C

*If temperature reading is outside the operating range, perform the following trouble shooting items.

1. If there is no obvious mechanical breakdown, take another temperature reading and record.

2. If the temperature is too low, adjust the controls up slightly and monitor frequently to prevent the contents from freezing.

3. Check the physical integrity and for continuous electrical power.

4. If the anomaly is a result of mechanical problems, move all samples to another refrigerator and notify affected lab areas.

5. If a minor adjustment will return the temperature to specification, adjust the temperature control and monitor the unit hourly to ensure the minor modification does not result in freezing the unit's contents.

6. Notate all actions in the logbook and unit problem report, notify area supervisor and the quality assurance area. If necessary the QA Supervisor will contact service representative the next business day:

Figure 35

D) Balance Monitoring

Each analytical and top loading balance used at the TriMatrix laboratory is monitored for accuracy. All daily checks are recorded in an TriMatrix balance log. Refer to section 4.3.3 below.

E) Standard Record Books

All standards and calibration solutions used at the TriMatrix laboratory are prepared when possible, from reagents or solutions traceable to national standards. Each standard solution, whether a stock, an intermediate or a working concentration is traceable to its origin source by means of standard logbooks.

Each standard logbook contains the following information:

- The analyte or analytes contained in the standard
- The concentration
- The solvent used to prepare the standard
- The preservative (i.e., nitric acid)
- The date of preparation
- Initials of the preparer
- The standard expiration date
- The standard reference number

The last item, the reference number, is a unique identifier which is an extension of the stock reagent identification number and any subsequent dilutions.

F) Pipet Logs

All autopipetors utilized within the TriMatrix laboratories for the delivery of standard solutions, dilutents, and reagents are periodically checked for delivery accuracy. Because these pipetors contain mechanical parts, they are subject to inaccuracies if not properly maintained and calibrated.

The TriMatrix laboratories perform monthly calibration checks on all autopipetors. All calibration activities are documented in an TriMatrix pipet logbook. Each log is identified by manufacturer name and model number, the pipetor serial number (if available), and the starting and ending dates encompassed. All complete pipet logbooks are assigned document control numbers, inventoried and properly archived.

G) QC Reports, Lists and Charts (Internal)

Quality control reports are used also within the TriMatrix laboratories to monitor the analytical process and to provide a means by which this analytical process can be viewed over time. These reports range from internal audit reports to management to standard control or SQC charts. It is the responsibility of the quality assurance staff to compile, monitor, and maintain the necessary quality control reports, which will allow both management and the laboratory staff the means to monitor the control of all analytical data.

Examples of efforts available for this monitoring process are presented in Figures 36 through 39.

TRIMATRIX LABORATORIES, INC. - GRR
Quality Assurance (Shewhart) Chart

Sample Duplicates (Percent Differences)
from 01-FEB-1995 to 25-JAN-1996

INORGANIC LABORATORY

Parameter: NITROGEN, NITRATE
Method: Colorimetric, Auto-Cadmium Reduction

Application: WATER
Reference: USEPA-353.2

ODL: 0.05
Unit: mg/l

Range: 1

Run Date	Oper	Sample	ZERO 0.0%	MEAN 1.3%	UWL 9.0%	UCL 13.0%
24-JAN-96	LEW	136506	**********			
24-JAN-96	LEW	136463	**********			
24-JAN-96	LEW	136441	**********			
16-JAN-96	LEW	135829	**********			
02-JAN-96	LEW	135062	**********			
27-DEC-95	LEW	135187	**********			
21-DEC-95	LEW	135013	**********			
21-DEC-95	LEW	135007	**********			
20-DEC-95	LEW	134835	**********			
15-DEC-95	LEW	134540	**********			
14-DEC-95	LEW	134298	**********			
13-DEC-95	LEW	133144	! **			
07-DEC-95	LEW	133654	**********			
07-DEC-95	LEW	133645	**********			
10-NOV-95	LEW	131728	**********			
10-NOV-95	LEW	131785	**********			
09-NOV-95	LEW	131530	**********			
02-NOV-95	LEW	130803	**********			
02-NOV-95	LEW	130794	**********			
01-NOV-95	LEW	130846	**********			
20-OCT-95	LEW	130404	**********			
25-OCT-95	LEW	130277	**********			
20-OCT-95	LEW	130130	**********			
13-OCT-95	LEW	129537	**********			
11-OCT-95	LEW	129149	**********			
07-OCT-95	LEW	129009	**********			
04-OCT-95	LEW	128811	**********			
29-SEP-95	LEW	128516	**********			
27-SEP-95	LEW	128307	**********			
21-SEP-95	LEW	127866	**********			
21-SEP-95	LEW	127435	**********			
20-SEP-95	LEW	127767	**********			
14-SEP-95	LEW	127165	**********			
13-SEP-95	LEW	126919	**********			
13-SEP-95	LEW	126599	**********			
07-SEP-95	LEW	126562	**********			
30-AUG-95	LEW	126113	**********			
11-AUG-95	LEW	124831	**********			
08-AUG-95	LEW	124347	**********			
27-JUL-95	LEW	123600	**********			
25-JUL-95	LEW	123586	**********			
20-JUL-95	LEW	123168	**********			
19-JUL-95	LEW	123264	**********			
19-JUL-95	LEW	123192	**********			

Figure 36

TRIMATRIX LABORATORIES, INC. - GRR
Quality Assurance Report

Laboratory Control Samples (Percent Recoveries)
from 01-FEB-1995 to 25-JAN-1996

Parameter: NITROGEN, NITRATE
Method: Colorimetric, Auto-Cadmium Reduction
Application: WATER
Reference: USEPA-353.2
ODL: 0.05
Unit: mg/l

Run Date	Analyst	Blank Conc.	Spike Amt.	Final Conc.	% Rec.
24-JAN-96	Laurel E. White	0	6.47	6.36	98.00
24-JAN-96	Laurel E. White	0	0.32	0.32	100.00
23-JAN-96	Laurel E. White	0	6.47	6.32	98.00
23-JAN-96	Laurel E. White	0	0.32	0.32	100.00
17-JAN-96	Laurel E. White	0	6.47	6.20	96.00
17-JAN-96	Laurel E. White	0	0.32	0.31	97.00
16-JAN-96	Laurel E. White	0	6.47	6.18	96.00
16-JAN-96	Laurel E. White	0	0.32	0.31	97.00
02-JAN-96	Laurel E. White	0	6.47	6.30	97.00
02-JAN-96	Laurel E. White	0	0.32	0.32	100.00
27-DEC-95	Laurel E. White	0	6.47	6.28	97.00
27-DEC-95	Laurel E. White	0	0.32	0.32	100.00
21-DEC-95	Laurel E. White	0	6.47	6.12	95.00
21-DEC-95	Laurel E. White	0	0.32	0.31	97.00
20-DEC-95	Laurel E. White	0	6.47	6.43	99.00
20-DEC-95	Laurel E. White	0	0.32	0.32	100.00
15-DEC-95	Laurel E. White	0	6.47	6.18	96.00
15-DEC-95	Laurel E. White	0	0.32	0.32	100.00
14-DEC-95	Laurel E. White	0	6.47	6.22	96.00
14-DEC-95	Laurel E. White	0	0.32	0.32	100.00
13-DEC-95	Laurel E. White	0	6.47	6.32	98.00
13-DEC-95	Laurel E. White	0	0.32	0.32	100.00
07-DEC-95	Laurel E. White	0	6.47	6.35	98.00
07-DEC-95	Laurel E. White	0	0.32	0.32	100.00
07-DEC-95	Laurel E. White	0	6.47	6.29	97.00
07-DEC-95	Laurel E. White	0	0.32	0.32	100.00
02-DEC-95	Laurel E. White	0	6.47	6.31	98.00
02-DEC-95	Laurel E. White	0	0.32	0.32	100.00
30-NOV-95	Laurel E. White	0	6.47	6.31	98.00
30-NOV-95	Laurel E. White	0	0.32	0.32	100.00
29-NOV-95	Laurel E. White	0	6.47	6.27	97.00
29-NOV-95	Laurel E. White	0	0.32	0.32	100.00
15-NOV-95	Laurel E. White	0	6.47	6.21	96.00
15-NOV-95	Laurel E. White	0	0.32	0.32	100.00
10-NOV-95	Laurel E. White	0	6.47	6.24	96.00
10-NOV-95	Laurel E. White	0	0.32	0.32	100.00
09-NOV-95	Laurel E. White	0	6.47	6.33	98.00
09-NOV-95	Laurel E. White	0	0.32	0.32	100.00
03-NOV-95	Laurel E. White	0	0.32	0.33	103.00
03-NOV-95	Laurel E. White	0	6.47	6.51	101.00
03-NOV-95	Laurel E. White	0	0.32	0.33	103.00
02-NOV-95	Laurel E. White	0	6.47	6.13	95.00
02-NOV-95	Laurel E. White	0	0.32	0.31	97.00
01-NOV-95	Laurel E. White	0	6.47	6.08	94.00
01-NOV-95	Laurel E. White	0	0.32	0.31	97.00
28-OCT-95	Laurel E. White	0	6.47	6.20	96.00
28-OCT-95	Laurel E. White	0	0.32	0.31	97.00

Figure 37

TRIMATRIX LABORATORIES, INC. - GRR
Quality Assurance (Shewhart) Chart

Laboratory Control Samples (Percent Recoveries)
from 01-FEB-1995 to 25-JAN-1996

INORGANIC LABORATORY

Parameter: NITROGEN, NITRATE
Method: Colorimetric, Auto-Cadmium Reduction

Application: WATER
Reference: USEPA-353.2

ODL: 0.05
Unit: mg/l

Run Date	Oper	LCL 84.0%	LWL 89.0%	MEAN 98.7%	UWL 108.0%	UCL 113.0%
24-JAN-96	LEW			**		
24-JAN-96	LEW				*	
23-JAN-96	LEW			**		
23-JAN-96	LEW				*	
17-JAN-96	LEW			*********		
17-JAN-96	LEW			******		
16-JAN-96	LEW			*********		
16-JAN-96	LEW			******		
02-JAN-96	LEW			******		
02-JAN-96	LEW				*	
27-DEC-95	LEW			******		
27-DEC-95	LEW				*	
21-DEC-95	LEW			**********		
21-DEC-95	LEW			******		
20-DEC-95	LEW				*	
20-DEC-95	LEW				*	
15-DEC-95	LEW			*********		
15-DEC-95	LEW				*	
14-DEC-95	LEW			*********		
14-DEC-95	LEW				*	
13-DEC-95	LEW			**		
13-DEC-95	LEW				*	
07-DEC-95	LEW			**		
07-DEC-95	LEW				*	
07-DEC-95	LEW			******		
07-DEC-95	LEW				*	
02-DEC-95	LEW			**		
02-DEC-95	LEW				*	
30-NOV-95	LEW			**		
30-NOV-95	LEW				*	
29-NOV-95	LEW			******		
29-NOV-95	LEW				*	
15-NOV-95	LEW			*********		
15-NOV-95	LEW				*	
10-NOV-95	LEW			*********		
10-NOV-95	LEW				*	
09-NOV-95	LEW			**		
09-NOV-95	LEW				*	
03-NOV-95	LEW				**********	
03-NOV-95	LEW				********	
03-NOV-95	LEW				**********	
02-NOV-95	LEW			**********		
02-NOV-95	LEW			******		
01-NOV-95	LEW			**********		

Figure 38

TRIMATRIA LABOR[illegible]IES, INC. - GRR
Quality Assurance (Shewhart) Chart

Matrix Spike Duplicates (Percent Differences)
from 01-FEB-1995 to 25-JAN-1996

METALS LABORATORY

Parameter: SILVER, TOTAL — Application: WW — ODL: 10
Method: Atomic Emission-ICP — Reference: EPA-200.7 — Unit: ug/l — Range: 2

Run Date	Oper	Sample	ZERO 0.0%	MEAN 3.6%	UWL 11.0%	UCL 20.0%
18-JAN-96	DWJ	136103		*******		
18-JAN-96	DWJ	135981		**********		
16-JAN-96	DWJ	135896		**********		
11-JAN-96	DWJ	135485	**********			
11-JAN-96	DWJ	135640	**********			
09-NOV-95	DWJ	131303	**********			
07-NOV-95	DWJ	131205	***			
13-OCT-95	DWJ	129347	**********			
12-OCT-95	DWJ	129099	**********			
29-SEP-95	DWJ	128408	********			
27-SEP-95	DWJ	128028	**********			
15-SEP-95	DWJ	126774	***			
29-AUG-95	DWJ	125787	**********			
24-AUG-95	DWJ	125597	***			
24-AUG-95	DWJ	125355	********			
04-AUG-95	DWJ	124173	********			
04-AUG-95	DWJ	124100	********			
04-AUG-95	DWJ	124012		**********		
04-AUG-95	DWJ	123833	********			
05-JUL-95	DWJ	121909	**********			
05-JUL-95	DWJ	121911	***			
24-JUN-95	DWJ	120732	********			
16-JUN-95	DWJ	120258	**********			
16-JUN-95	DWJ	120257	***			
16-JUN-95	DWJ	120179	**********			
13-JUN-95	DWJ	119354	********			
13-JUN-95	DWJ	119841		**********		
22-MAY-95	DWJ	117741		**		
28-APR-95	DWJ	115465		**********		
28-APR-95	DWJ	115642	********			
24-APR-95	DWJ	115326	**********			
11-APR-95	DWJ	114171	********			
10-APR-95	DWJ	114301	********			
10-APR-95	DWJ	114320		**********		
06-MAR-95	DWJ	111627	********			
16-FEB-95	DWJ	110544		**********		
01-FEB-95	DWJ	109505		**********		
01-FEB-95	DWJ	109460		**********		

Figure 39

Quality control reports are used extensively in the TriMatrix laboratory to access the analytical process. All QC reports are created through the TriMatrix LIMS system. Many of these reports such as the analytical and QC batch detail reports are utilized daily to monitor all aspects of quality control, i.e., method accuracy, precision, completeness and provide the means for overall data assessment at the batch level.

Many options are available through the TriMatrix LIMS in creating each type of report.

Presented in Table 3 is a list of quality control reports provided through our LIMS and a brief summary or description of their use.

TABLE 3

Report Name	Description of Use
Analytical Batch Detail Report	This report displays all analyses for a given analytical batch, including all sample data and quality control checks as tested in the analytical batch.
QC Batch Detail Report	This report displays all results for a given QC or matrix batch. The samples may have been analyzed in several analytical batches, but are presented as a review of the extraction or pretreatment batch.
Results List	This report generally is utilized in conjunction with a Shewart Chart to display QC data in a tabular form.
Shewart Chart	The Shewart report is created to graphically display the distribution of QC data. This report is available to display the distribution of both accuracy and precision data.
Compliance Summary	The compliance summary report was created to display batch and method compliance or completeness as defined for each analytical protocol. Also displayed in this report is a number of warning and control failures with specific data presented for all out-of-control data points.
Biased Statistics	The biased statistics report is utilized in listing tests which have become biased by having 7 continuous results on one side of the mean.
Control Limit Comparison Report	The control limit comparison report was designed to present current in-house statistical windows in comparison to those published in a specific method.

Statistics Summary	This report lists current statistical values by test for a given lab area. The information presented includes: no. of observations, mean, standard deviation, upper and lower control and warning limits and method specific or _____ limits.
Statistics Report by Class	This report is designed to present operational statistical windows for designated QC types and test lists.
Deviant Statistics	The deviant statistics report is used to list tests that have statistical values which have deviated significantly from the previous statistical data.
Locked Statistics	This report displays all locked statistics for a given laboratory area. An example of a locked statistic is an ICV or initial calibration verification standard for ICP analysis.
Analyst Certifications	The analyst certification report is produced to review laboratory control sample data for a given analyst, test, and defined time period. This report is utilized to review an analyst's performance.
Pre-Defined Qualifier Report	This report list defined all analysis, sample and batch qualifiers that are available for data qualifications. The report is used for reference purposes only.

Examples of these reports are presented in Appendix C.

4.1.2.10 Confidentiality and Proprietary Rights

Since a significant amount of information may be received in the laboratory by telephone, written submittals, and personal contact regarding details of a client's operations, it is essential that strict confidentiality be maintained in the handling of all client details. Client data is to be protected in locked filing cabinets and in limited access computer files. Under no circumstances is the name of a client or any information regarding that client to be revealed to another client or to a regulatory agency without the client's written permission.

Any details of a client's operations which have necessarily been revealed to the laboratory for testing purposes are to be considered as proprietary and protected by patents, copyrights, infringement laws, or other legal constraints against disclosure.

4.1.2.11 Document Storage and Traceability

Archiving of information at the TriMatrix laboratories has been designed to meet both short-term and long-term storage needs. Archives are maintained for a wide variety of data and documentation. These archives can be categorized into two main groups, a) document archives (physical documents) and b)electronic archives (data files). The following Table 4 illustrates the current TriMatrix archival systems, their location and duration.

TABLE 4
DATA ARCHIVING SYSTEMS

DOCUMENT ARCHIVES

Document Description	Storage Location	Storage Duration	Optically Scanned
Laboratory benchsheets	on-site	1 year	yes
Laboratory benchsheets	off-site	6 years	yes
Instrument Print-Outs (raw data)	on-site	1 year	yes[1]
Instrument Print-Outs (raw data)	off-site	6 years	yes[1]
Laboratory Logs (run, maint., etc,)	on-site	3 years	yes
Laboratory Logs (run, maint., etc,)	off-site	6 years	yes
Client Files (reports, correspondence, invoices)	on-site	1 year	yes[2]
Client Files (reports, correspondence, invoices)	off-site	6 years	yes[2]
Proposal Files	on-site	5 years	no
Purchase Agreements	on-site	5 years	no

1. Instrument printouts are only scanned if they contain handwritten notations. All other data is stored in both the hard copy version and as an electronic file (see below)
2. All LIMS generated client/invoice reports are automatically archived to the optical disk system.

ELECTRONIC ARCHIVES

File Description	Storage Location	Storage Duration	Storage Media
Instrument Data Files-GC/MS(Extrels)	on-site	3-5 years[3]	Tape
Instrument Data Files-GC/MS(ION Traps)	on-site	10 years	Optical Disk
Instrument Data files-GC (TurboChrome)	on-site	10 years	Optical Disk
Instrument Data files-AA/ICP	on-site	10 years	Optical Disk
Instrument Data files-Auto Analyzer	on-site	10 years	Optical Disk

3. All tapes are retained indefinitely, the anticipated life is expected to be 3-5 years.

Documentation records or logs are maintained for all archival systems to aid in the quick retrieval of information. Extended archival periods or special procedures are also in place for some projects and clients.

4.1.3 Standard Operating Procedures (SOPs)

A standard operating procedure (SOP) is a guide that addresses the need for each laboratory to maintain a manual that clearly defines the exact steps to be followed in performing an analytical procedure.

This delineation of these exact steps will improve the confidence and the ability to reproduce the analytical conditions, which in turn will help the overall reproduction of analytical data.

A significant part of the variability in the analytical data generated by a given method is in part largely due to minor variations in the operation and/or inability to routinely reproduce the steps in the analytical process. A standard operating procedure should clearly define these steps and help minimize these minor variations.

Many of the methods published today by various agencies provide only general guidance in performing an analytical determination. This is another important reason why specific laboratory procedures are needed.

All SOP's developed by the TriMatrix laboratories are subject to a review process where signatures or approvals are required from the appropriate area supervisor, the quality assurance supervisor and the laboratory manager. In addition to this overall approval process, each page of an SOP is individually approved by the laboratory area and the quality assurance supervisors (see enclosed Figures 40 and 41).

4.1.3.3 SOP Documentation and Control

All SOPs are assigned procedure numbers that are unique to each laboratory, the effective date, revision number, information on the author, total number of pages and identification of any individual page revisions.

All TriMatrix SOPs are controlled through a controlled document management system. The system is used to document the release of any SOP to in-house staff, external clients or government agencies. This documentation procedure is maintained on an TriMatrix standard operating procedure inventory log form (see enclosed Figure 42). This form is generated for each SOP and revision number. All forms are maintained in a locked SOP file. Entries are made for each copy produced, including date issued, by whom, name of chemist, lab area, client, etc. to whom the SOP was issued and a unique document control number.

When an SOP is revised, all previously issued versions must be collected before replacement with the latest revision.

All copied SOPs are printed on special paper that is clearly labeled as a controlled TriMatrix document (see attached Figure 43).

4.1.3.1 SOP Categories-Types

The TriMatrix laboratories standard operating procedures are written for almost all laboratory activities. The categories utilized in the organization of SOPs are presented in Table 5:

TABLE 5

Trace Metals	Instrumental-General
Gas Chromatograph	Gas Chromatography/Mass Spectroscopy
Spectrophotometric Procedures	Titrimetric Procedures
Gravimetric Procedures	Electrochemical/Potentiometric Procedures
Extractions-Organic	Quality Assurance
Sales and Customer Service	Business and Accounting
Laboratory Computer Operations	Laboratory Safety and Security
Sample Receiving, Storage, & Disposal	Miscellaneous
Bottle Prep	Inorganic-General
Microbiology	
Waste Characterization	

4.1.3.2 SOP Development, Formatting and Review

All standard operating procedures are developed and written to the specifications outlined in the TriMatrix guidelines for the preparation of an SOP. These guidelines are presented as a standard operating procedure and are available in different formats that have been designed to accommodate analytical tests, non-tests such as extractions or digestions, and a documentation or non-analytical activity. The guidelines were developed from both USEPA and ASTM protocols for the creation of a standard operating procedure.

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

CYANIDE, TOTAL

PRELIMINARY MACRODISTILLATION

USEPA METHOD #SDM 4500-CN B, 4500 CN C

APPROVALS:

Inorganic Supervisor:_____ Date:_____

QA/QC Supervisor:_____ Date:_____

Laboratory Manager:_____ Date:_____

Procedure Number:

Revision Number: 0.0 By: Betty Doyle Effective Date: 2/8/95

Total Number of Pages: 10 Pages Revised: None

Figure 40

Subject: Cyanide, Total
Preliminary Distillation

Procedure No:
Revision No: 0.0
Effective Date: 2/8/95

USEPA Method SDM 4500-CN B, 4500-CN C

1.0 PRINCIPAL METHOD REFERENCE

Standard Methods for the Examination of Water and Wastewater, 18th Edition (1992), 4500-CN B, 4500-CN C.

2.0 PARAMETER LIST

Cyanide, Total.

3.0 SCOPE AND APPLICATION

3.1 This method is applicable to the determination of cyanide in soil, sludge, and waste. This method may be used for water and wastewater, if needed, using 250 mls of sample and 250 mls of DI H_2O for distilling.

3.2 The method detects inorganic cyanides that are present as either simple soluble salts or complex radicals.

3.3 The applicable range for this method is 0.05 to 10 mg/kg.

3.4 The reportable detection limit for this method is 0.05 mg/kg but varies as to % solids for soils and sludges. Wastes are reported on an "as received" basis.

4.0 SUMMARY OF TEST METHOD

The cyanide present in the sample is released as a gas (HCN) upon the addition of sulfuric acid. The HCN is collected in a NaOH scrubbing solution. The NaOH scrubbing solution is then analyzed for cyanide using EPA Method 335.3/9012. This method has been modified: Bismuth nitrate is used to remove sulfide interference rather than lead carbonate.

Figure 41

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURES
NUMBER LOG
MASTER SECTION REFERENCE

Section Number	Section Titles
0 1	Trace Metals
0 2	Instrumental - General
0 3	Gas Chromatography
0 4	Gas Chromatography/Mass Spectroscopy (GC/MS)
0 5	Spectrophotometric Procedures
0 6	Titrimetric - Procedures
0 7	Gravimetric - Procedures
0 8	Electrochemical/Potentiometric - Procedures
0 9	Extractions - Organic
1 0	Quality Assurance
1 1	Sales and Customer Service
1 2	Business and Accounting
1 3	Laboratory Computer Systems
1 4	Laboratory Safety and Security
1 5	Sample Receiving, Storage, Disposal and Bottle Prep.
1 6	Miscellaneous
1 7	Microbiology
1 8	Inorganic - General
1 9	Waste Characterization
	Prefixes: GR- Grand Rapids, LI - Livonia
	Example: Procedure Number GR-15-104, "Hazard Waste Disposal" for Grand Rapids Lab.

Figure 42

4.1.4 LIMS Validation Procedures

The TriMatrix laboratory has developed and maintained a custom laboratory information system (LIMS). This TriMatrix system was exclusively designed to encompass all aspects of our laboratory operations. The main functions of the TriMatrix LIMS are:

- Project Management
- Sample Management
- Work Scheduling and Management
- Data Entry, Verification and Approval
- Report Generation
- Invoicing

4.1.4.1 Good Automated Laboratory Practices (GALP) Compliance

In designing the TriMatrix LIMS system, certain fundamental approaches were taken in an effort to maintain proper authority, documentation and control during the design, programming and implementation stages of the system.

Recently, the TriMatrix system has been reviewed against the USEPA's publication "Good Automated Laboratory Practices". Our review against the guidelines presented in the manual has validated our initial approach to the processes involved with the TriMatrix LIMS.

The GALP guidelines emphasize the need for proper organization and personnel, the correct facility to maintain system integrity, obtain the proper equipment to maximize on-going operations, identification and maintenance of system security, data security, consistent system use, software documentation/maintenance, operational logs and backup/recovery schemes.

The TriMatrix laboratory has numerous in depth protocols to deal with these guidelines as well as many others. Many of the procedures utilized to describe our efforts to meet these guidelines are presented in a series of standard operating procedures which includes the following categories and descriptions:

Security — Describe how the automated data collection system is kept secure. Discuss physical security of the computer(s), access to use of the installed software, ability to modify the installed software, ability to install additional software, and access to the data (*i.e.* retrieval, insertion, modification, and deletion).

Raw Data — Define what constitutes raw data (vs. processed data).

Data Entry — Describe how data, both raw and processed, is entered into the data storage files, and how the operator entering the data is identified in the files.

Verification — Describe how the accuracy of the data, whether entered manually or automatically, is verified.

Errors — Identify all anticipated error codes & messages, who is responsible for taking corrective action, and what corrective action should be taken.

Processing — Describe how the raw data is processed (manipulated) and analyzed (interpreted).

Change Control — Describe the ways in which data may be changed, how the original data is preserved for the historical record, and how the changed data is documented (*i.e.* who made the change, when, and why).

Reporting — Describe how the integrity of the data is protected on all reports, whether visual hardcopy or magnetic media.

Backup and Recovery — Describe the schedule and method of backing up the data files, how the backups are logged (manually or automatically), and how to recover/restore data from a backup.

Archiving & Purging — Describe how data is migrated to intermediate- and long-term storage, and how data is retrieved from that storage.

Maintenance — Describe the procedures for maintaining the hardware used in the collection, processing, reporting, backup, and archival of the data.

The TriMatrix laboratory will continue to monitor and implement procedures and protocols in an effort to meet or exceed the GALP requirements.

4.2 Sample Control, Flow and Storage

Presented in the following section is a description of the policies and procedures that were developed to identify, monitor and document the flow of samples through the TriMatrix Laboratory flow chart depicting this process is presented in Figure 44.

4.2.1 Project Initiation

When samples are received at the TriMatrix laboratories, the necessary information that will direct the analytical scheme has all ready been developed and implemented within the TriMatrix project initiation/project management process. This process starts with the award of a contract or proposal, a client request or a pre-scheduled sampling event. The basic steps and supporting documentation involved in the project initiation process begins with the gathering of project information, communications with all affected laboratory areas and the input of required project related data into the LIMS system.

If a new project will require support from the analytical facilities, that project must be coordinated with the laboratory supervisors and the laboratory manager prior to project pricing and sample receipt. Routine samples are those samples and analyses which are continuously processed by TriMatrix.

Projects which are non-routine are those that may require special testing, or which request parameters not routinely run within the laboratory, special holding times, or rush turnaround. Non-routine projects will require approval from all affected laboratory areas. This approval process is communicated in several different ways, including everything from the signing of a quality assurance project plan (QAPP) to the transmission and receipt of an electronic mail message.

The development of a project within the laboratory also involves the preparation and shipment of sample collection materials and containers. The processes involved in the procurement, preparation and shipment of sample collection materials and bottles are presented below.

TriMatrix Laboratories, Inc.

Sample Flow Diagram

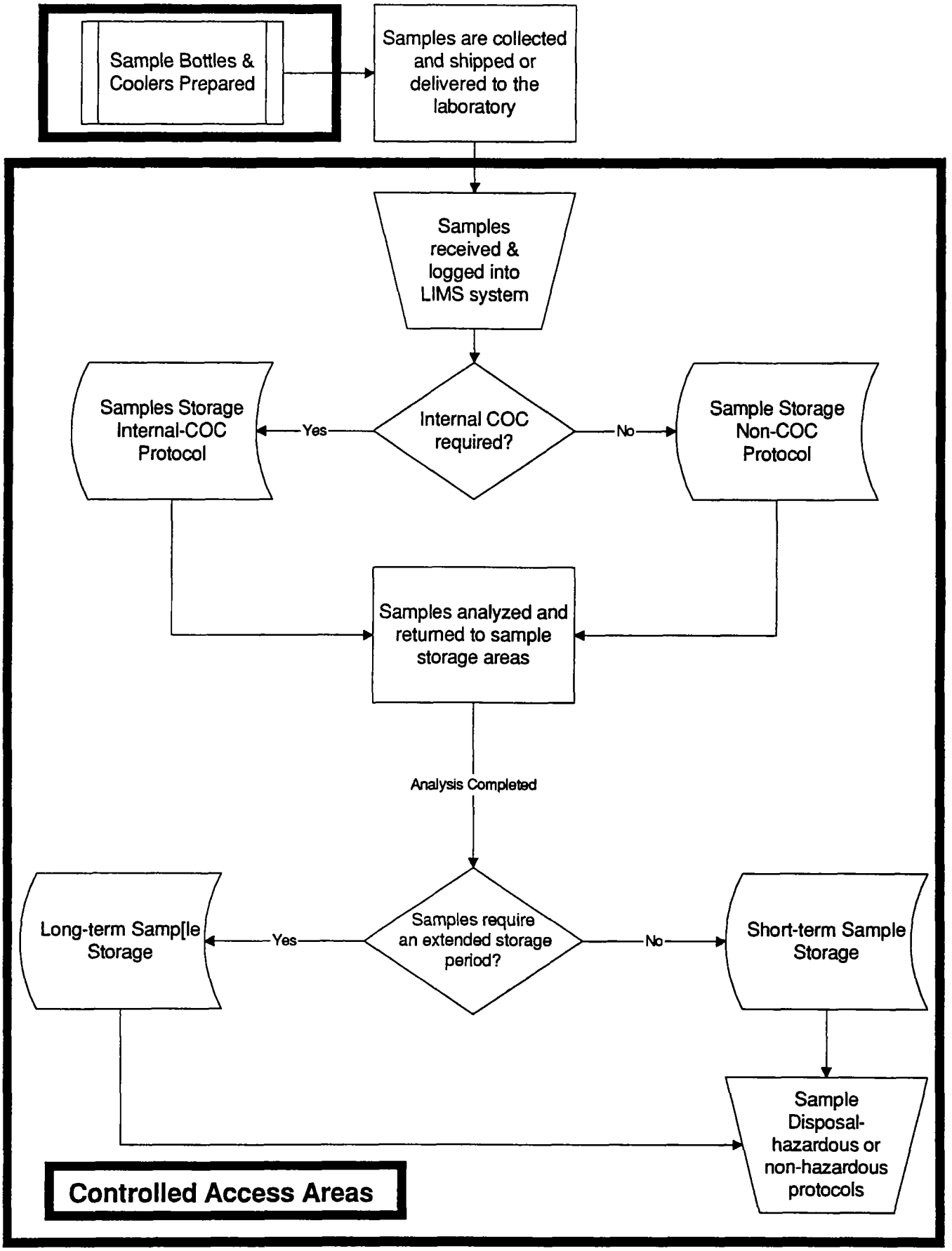


Figure 44

4.2.1.1 Sample Containers and Materials Procurement

The TriMatrix laboratories utilize only virgin bottle ware for all sample collection kits. Plastic ware is typically Nalge brand, NDPE, while glassware is I-CHEM Series 200 (or equivalent) pre-cleaned with the exception of oil and grease and TOX containers. These bottle types require special handling and cleaning prior to shipment.

I-CHEM Series 300 (or equivalent) bottle ware is available upon request and generally at an additional cost.

4.2.1.2 Preparation of Containers

All sample containers utilized for the collection and preservation of environmental samples are prepared by the TriMatrix bottle prep group. The staff members of this group focus the activities exclusively in the area of sample container procurement, preparation, and shipping. The procedures utilized performing these activities are outlined in the TriMatrix SOP for bottle preparation.

The TriMatrix laboratories have developed a unique color coded bottle tagging system for the purposes of defining and differentiating the various sample bottle types and the chemical additives that are required for proper sample preservation.

In conjunction with our color coded tagging system, a form was developed to display the coding system, identify chemical preservatives and provide a means of listing the exact quantity, bottle type and preservatives required for each sample location in a tabular format. An example of the TriMatrix "sample inventory and master bottle pricing list" form is provided in Figure 13 in section 4.1.2.1 of this document. An example of the TriMatrix sample bottle tagging system was presented in Figure 19 in Section 4.1.2.2. This illustration shows both

sides (front and back) of a bottle tag as it appears on a prepared sample container, upon completion of the log-in process.

Quality assurance measures utilized in monitoring sample coolers includes the use of trip blanks for volatile organics and temperature blank bottles which travel with all level 3 and higher projects. Trip blanks for all container types shipped are available upon request.

4.2.1.3 Sample Container Shipment

When all containers have been assembled as requested on the "master bottle packing list", the bottles are then packaged and placed into one or more shipping coolers. Each cooler is packed with bottles that have been carefully enclosed in poly bubble pack. 40 ml glass vials are packed in small bubble pack bags.

Coolers are generally organized in such a way to help minimize time spent in the field. This is usually accomplished by packing sets of bottles together that will be required at a particular sample location. A copy of the bottle pricing list is placed in each cooler with the sample locations highlighted to indicate which bottles are contained within the cooler. This practice is usually only necessary when sample collection sites require a different series of containers.

Also presented in each cooler in addition to the bottle packing list is a series of instructions or comments about the containers, material safety data sheets for all chemical preservatives present, a return address label, an external COC form, and if required TriMatrix sample bottle custody seals. All materials are packaged in a waterproof zip-lock type bag.

Examples of these additional materials are presented in Figures 45 to 48.

All sample coolers are sealed with a signed TriMatrix custody seal to validate the integrity of the containers at the sample site.

TriMatrix Laboratories, Inc.

PROJECT NOTES

Enclosed are the sample containers that are required for your project. We have included several project notes that will allow the safe handling of containers and allow the completion of your project in a timely manner.

- The containers are pre-cleaned; therefore no additional cleaning is required.
- Some sample containers contain preservatives; please do not rise or overfill. These preservatives are there to adhere to EPA set standards.
- The chemicals in the containers are hazardous. Extreme caution should be used when handling. The sampler should not breathe or come in physical contact with these chemicals. For your safety, please read the complete Material Safety Data Sheets that are enclosed.
- When doing soil sampling, please clean off any residual soil from the outside of the coolers. This can prevent contamination of other samples in the cooler.
- Please fill out the sample identification tags as completely as possible.
- Please fill out the enclosed chain of custody form for adequate sample tracking.

If you have any questions concerning your sampling, please phone TriMatrix at (616) 975-4500 and ask for one of the Laboratory Client Services personnel.

TriMatrix Laboratories, Inc.
Laboratory Client Services

Figure 45

NAC

NITRIC ACID

[redacted]

Watery liquid Colorless to light brown Choking odor

Sinks and mixes with water. Harmful vapor is produced.

AVOID CONTACT WITH LIQUID AND VAPOR. Keep person away.
Wear chemical protective suit with self-contained breathing apparatus.
Stop discharge if possible.
Isolate and remove discharged material.
Notify local health and pollution control agencies.

Fire

Not flammable.
May cause fire on contact with combustibles.
Flammable gas may be formed on contact with metals.
Poisonous gases are produced when heated.
Wear chemical protective suit with self-contained breathing apparatus.
Cool exposed containers with water.

Exposure

CALL FOR MEDICAL AID.

VAPOR
Will burn eyes, nose and throat.
If inhaled, will cause difficult breathing or loss of consciousness.
Move to fresh air.
If breathing has stopped, give artificial respiration.
If breathing is difficult, give oxygen.

LIQUID
Will burn skin and eyes.
Harmful if swallowed.
Remove contaminated clothing and shoes.
Flush affected areas with plenty of water.
IF IN EYES, hold eyelids open and flush with plenty of water.
IF SWALLOWED and victim is CONSCIOUS, have victim drink water or milk.
DO NOT INDUCE VOMITING.

Water Pollution

HARMFUL TO AQUATIC LIFE IN VERY LOW CONCENTRATIONS.
May be dangerous if it enters water intakes.
Notify local health and wildlife officials.
Notify operators of nearby water intakes.

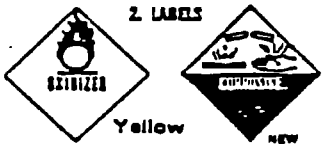
[redacted]

1. RESPONSE TO DISCHARGE

[redacted]

Issue warning—corrosive
Restrict access
Evacuate area
Dissolve and flush

2. LABELS



3. CHEMICAL DESIGNATIONS

3.1 Synonyms: No common synonyms
3.2 Coast Guard Compatibility Classification: Nitric acid
3.3 Chemical Formula: $HNO_3 \cdot H_2O$
3.4 IMCO/United Nations Numerical Designation: 8.0/2031

4. OBSERVABLE CHARACTERISTICS

4.1 Physical State (as shipped): Liquid
4.2 Color: Colorless
4.3 Odor: Acrid; sweet to acrid

5. HEALTH HAZARDS

5.1 Personal Protective Equipment: Air mask; rubber acid suit, hood, boots and gloves; chemical goggles; safety shower and eye bath.

5.2 Symptoms Following Exposure: Vapor irritates eyes and respiratory tract; lung injury may not become apparent for several hours following exposure. Liquid may cause severe burns to eyes and skin.

5.3 Treatment for Exposure: INHALATION: remove to fresh air, administer artificial respiration if required. INGESTION: drink large volumes of water; do NOT induce vomiting. SKIN OR EYES: flush with water for at least 15 min.

5.4 Toxicity by Inhalation (Threshold Limit Value): 2 ppm

5.5 Short-Term Inhalation Limits: 15 ppm for 5 min.

5.6 Toxicity by Ingestion: Grade 3; LD_{50} 50 to 500 mg/kg

5.7 Late Toxicity: None

5.8 Vapor (Gas) Irritant Characteristics: 38-46%: Vapor is moderately irritating such that personnel will not usually tolerate moderate or high vapor concentrations. 95% Vapors cause severe irritation of eye and throat and can cause eye and lung injury. They cannot be tolerated even at low concentrations.

5.9 Liquid or Solid Irritant Characteristics: Severe skin irritant. Causes second- and third-degree burns on short contact and is very dangerous to the eyes.

5.10 Odor Threshold: Data not available

6. FIRE HAZARDS

6.1 Flash Point: Not flammable
6.2 Flammable Limits in Air: Not flammable
6.3 Fire Extinguishing Agents: Use water on adjacent fires.
6.4 Fire Extinguishing Agents Not to be Used: Not pertinent
6.5 Special Hazards of Combustion Products: May give off poisonous oxides of nitrogen and acid fumes when heated in fire.
6.6 Behavior in Fire: Decomposes and gives off poisonous oxides of nitrogen.
6.7 Ignition Temperature: Not flammable
6.8 Electrical Hazard: Not pertinent
6.9 Burning Rate: Not flammable

7. CHEMICAL REACTIVITY

7.1 Reactivity with Water: May react violently on mixing, but explosion or formation of vapor unlikely.
7.2 Reactivity with Common Materials: Very corrosive to wood, paper, cloth and most metals. Toxic red oxides of nitrogen are formed.
7.3 Stability During Transport: When heated may give off toxic red oxides of nitrogen.
7.4 Neutralizing Agents for Acids and Caustics: Flush with water.
7.5 Polymerization: Not pertinent
7.6 Inhibitor of Polymerization: Not pertinent

8. WATER POLLUTION

8.1 Aquatic Toxicity: 72 ppm/96 hr/mosquito fish/TL_m/fresh water
330-1000 ppm/48 hr/oysters/LC_{50}/salt water
8.2 Waterfowl Toxicity: Data not available
8.3 Biological Oxygen Demand (BOD): None
8.4 Food Chain Concentration Potential: None

9. SELECTED MANUFACTURERS

1. Allied Chemical Corp.
   Agricultural Division
   Morristown, N. J. 07960
2. E. I. duPont de Nemours & Co., Inc.
   Explosives Dept.
   Wilmington, Del. 19898
3. Hercules, Inc.
   Explosives & Chemical Properties Dept.
   Bessemer, Ala. 35020

10. SHIPPING INFORMATION

10.1 Grades or Purity: Various grades: 52-96%
10.2 Storage Temperature: Ambient
10.3 Inert Atmosphere: No requirement
10.4 Venting: Open or pressure-vacuum

11. HAZARD ASSESSMENT CODE

[redacted]

A-P

12. HAZARD CLASSIFICATIONS

12.1 Code of Federal Regulations: Oxidizer

12.2 NAS Hazard Rating for Bulk Water Transportation:

Category	Rating
Fire	0
Health	
Vapor Irritant	3
Liquid or Solid Irritant	4
Poisons	3
Water Pollution	
Human Toxicity	3
Aquatic Toxicity	3
Aesthetic Effect	2
Reactivity	
Other Chemicals	4
Water	0
Self-Reaction	0

12.3 NFPA Hazard Classifications:

Category	Classification
Health Hazard (Blue)	3
Flammability (Red)	0
Reactivity (Yellow)	0
	oxy

13. PHYSICAL AND CHEMICAL PROPERTIES

13.1 Physical State at 15°C and 1 atm: Liquid
13.2 Molecular Weight: Not pertinent
13.3 Boiling Point at 1 atm: 192.0°F = 88.9°C = 362.1°K
13.4 Freezing Point: -50°F = -45.6°C = 227.6°K
13.5 Critical Temperature: Not pertinent
13.6 Critical Pressure: Not pertinent
13.7 Specific Gravity: 1.49 at 20°C (liquid)
13.8 Liquid Surface Tension: Not pertinent
13.9 Liquid-Water Interfacial Tension: Not pertinent
13.10 Vapor (Gas) Specific Gravity: Not pertinent
13.11 Ratio of Specific Heats of Vapor (Gas): (est.) 1.248
13.12 Latent Heat of Vaporization: 214 Btu/lb = 119 cal/g = 4.98 × 10^5 J/kg
13.13 Heat of Combustion: Not pertinent
13.14 Heat of Decomposition: Not pertinent
13.15 Heat of Solution: -205 Btu/lb = -114 cal/g = -4.76 × 10^5 J/kg
13.16 Heat of Polymerization: Not pertinent

NOTES

REVISED 1978

Figure 46

TriMatrix
Laboratories, Inc.

5555 Glenwood Hills Parkway SE • PO Box 888692 • Grand Rapids, MI 49588-8692

Chain of Custody Record

COC No.
27036

Project Manager	Project Name		**For Lab Use Only**
Project No.	Sampler (Print)		Rack/Tray No:
	Sampler Signature		Lab Project #

Date Sampled	Time Sampled	Matrix*	Composite	Grab	Sample Identification	No. of Containers	No's Correspond to Bottle Packing List Container Type	Analysis Required/Comments	Sample No.	Filtered Date/Time
							1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20			
							1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20			
							1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20			
							1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20			
							1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20			
							1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20			
							1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20			
							1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20			
							1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20			

Relinquished By:	Date/Time	Received By:	Received to Lab By:	Date/Time	Logged in By:	Date/Time

* Matrix: Water ([illegible] astewater (WW), Soil (SOIL), Sludge (SLG), Air, Oil, Waste (WASTE)

F[illegible]re 47

Customer
Return Address
Label

FROM:

_____

_____

_____

Environmental Samples Enclosed

TO:

TriMatrix Laboratories, Inc.

5555 Glenwood Hills Parkway SE
PO Box 888692
Grand Rapids MI 49588-8692

Figure 48

4.2.2 General Laboratory Security

Access to the TriMatrix laboratory is handled in a secured fashion, where access is restricted to authorized personnel only. All laboratory areas including, sample storage, sample container preparation, analytical laboratories, sample preparation, sample disposal, analytical documents and clients files are secured by a keyless entry system. Non-authorized personnel may enter these areas if escorted by a laboratory staff member.

It is the responsibility of all laboratory staff members to insure that the rules of restricted access are followed and maintained at all times.

4.2.3 Sample Log-In

All samples received by TriMatrix must be logged in before any analyses are conducted. The purpose of the log-in procedure, including sequential numbering of all samples received, is to insure that TriMatrix has a means by which samples can be tracked, data can be stored, and quality control can be tracked for any sequence of events during a particular analytical period. The primary steps involved in the sample log-in process are presented below.

4.2.3.1 Sample Receipt/Examination

Examination of Shipping Container

Immediately upon receipt of a sample shipment at the TriMatrix Laboratory, the SC will examine the shipping container to ascertain and document the condition of the samples and to process Chain-of-Custody papers, where appropriate. The SC will record the condition of the shipping container, the identification of the shipper, the presence or absence of any seals on the container and the labeling which may include special instructions prior to opening the container. If the shipping container is damaged, a report will be sent immediately to the shipper and the lab supervisor (see Figure 17 Problem Submittal Report).

Samples received at the TriMatrix laboratories are required to be accompanied by an TriMatrix laboratory Chain-of-Custody (COC) form. For these samples received without this form, the SC will initiate the COC process. Additional information and an example of the TriMatrix COC form is presented in section 4.2.4 of this manual.

Unless the shipping container contents are marked "hazardous" the SC will proceed to open the sample container. If the SC had not previously identified the LIMS submittal appropriate for these samples, the SC will attempt to ascertain immediately the origin of the samples found in this container and contact the appropriate project chemist. If a submittal and the project chemist are not available, the SC will lock up the samples and notify the lab manager. The SC will identify whether or not all the samples have arrived intact, whether or not the labels are intact and attached properly, and whether or not the samples have leaked in any fashion. The SC will also identify any shipping instructions, field instructions, or any other materials that may be present in the shipping container.

Should the SC identify a submittal or delivery group as an internal COC project, the SC will initiate the procedures outlined in the TriMatrix standard operation procedure for internal sample custody and those procedures outlined in Section 4.2.4 of this manual.

4.2.3.2 Computer Log-In

All samples received at the TriMatrix laboratory are logged into the TriMatrix LIMS System by the sample coordinator. The LIMS system assigns a sequential number to every sample entered into the system. In conjunction to the sample identification number, the submittal or delivery group can be identified by a unique number which consists of the LIMS project number and a sequenced number of the delivery group.

4.2.3.3 Project and Sample Verification

The SC having opened the shipping container(s) and having examined all the samples will then verify that the LIMS project report (see previous Figure 14) matches the samples, the number of samples received, and is consistent with the requirements of the project and submittal as created within the TriMatrix LIMS system. If the samples are not consistent with the information presented in the project report, the SC will complete an TriMatrix non-conformance report and turn it over to the appropriate Project Chemist.

4.2.3.4 Sample Distribution

The TriMatrix Sample Coordinator will inspect to insure that all samples received at the TriMatrix facility are received in the appropriate containers with the correct preservatives (Samples which must be split at log-in to added error).

Bottles and Preservative Requirements

The TriMatrix Laboratory has a series of bottle and preservative requirements that must be met before the log-in of samples into the laboratory. In the event that TriMatrix is unable to provide sample bottles, or circumstances prevent the splitting of samples in the field, the SC will provide sample splitting services. These services will include taking the sample as received and subsampling it into the appropriate bottle and preservative requirements as set forward in Appendix D - Bottle and Preservative Requirements.

A. Sample Splitting-Water Samples

The SC will insure that sufficient sample volume is available before initiating the splitting of a sample. If uncertain, the SC will involve the laboratory supervisors in order to insure that all

as a problem and will notify the Project Chemist immediately for resolution.

When a bulk sample arrives for organic/inorganic analysis and sufficient sample exists, the SC will transfer the sample to the organic preparation supervisor who will split the organic aliquot and return all aliquots to the SC. The remaining sample will then be returned to the SC who will split off the inorganic aliquots into the proper preserved containers.

B. Sample Splitting-Solid Samples

When solid samples, such as sediment or soil, are to be received at TriMatrix, every attempt will be made by the Project Chemist and field sampling personnel to insure that two samples are provided as replicates for the appropriate tests. One of these samples will be assigned to the organic facility; the other will be assigned to the inorganics facility. If only the sample is received and if organic analyses are required, the organics preparation chemist will be responsible for the initial splitting of the sample. Solid samples will be made homogeneous by either one or all of the following manners:

- Sitrring
- Grinding
- Particle separation (sieving)
- Quartering by ASTM Procedures

The lab area supervisor and the SC are responsible for the decisions on how a solid sample will be split. Problems or concerns which may arise on a solid sample will be addressed to the Project Chemist and the laboratory manager for resolution. After the organic portions have been removed or split, the remaining sample will be provided to the inorganic facilities for any further splitting they deem necessary.

C. Sample Identification/Labeling

All samples received at the TriMatrix laboratories are labeled by the SC at the time of log-in. These labels include the necessary information for proper identification of not only the sample ID number but also contain information on any potential for flammability, reactivity, contact or health based risks.

In addition to the sample identification tagging, all TriMatrix bottle and preservative types are clearly identified by means of a color coded tagging system. (As presented in section 4.2.1.1 "Sample Containers and Materials Procurement"). This section allows everyone involved in the analytical process from sample collection, sample analysis and sample disposal to clearly identify all containers for their intended use and chemical preservatives. This color coded process helps insure the right container type and preservative is utilized for the requested analytical procedure.

D. Sample Storage

- The SC, after completing all the log-in process of various samples connected with a particular project, will store the samples in the designated areas in the TriMatrix laboratory.

- Routine Water and Solid Samples: Samples which need to be refrigerated will be stored in the walk in facility designated for all routine water and soil samples.

- Routine Volatile Water and Solid Samples: All these samples are placed in the designated VOA refrigerator(s) located within the analytical facility. Volatile water and soil samples are segregated and stored separately. No other samples or standards may be stored in the VOA refrigerator(s).

- Routine Water and Solid Samples for Metal Parameters: The preserved water samples and solid samples, which are not preserved, may be stored on shelves designated for the metals analysis.

- Odoriferous and Hazardous Samples: These samples are stored in a special vented facility within the laboratory which is designated for odoriferous and hazardous samples. These samples will be identified to the laboratory by means of a sample or submittal narrative within the TriMatrix LIMS System.

All samples that are involved as physical evidence in a legal procedure or simply identified as Chain-of-Custody will be handled under certain procedural safeguards.

All samples of this nature will be stored within the locked confines of the analytical laboratory. Access is only available to authorized personnel.

4.2.4 Chain-of-Custody (COC)

All samples received at the TriMatrix laboratory will require some form of chain-of-custody (COC). The TriMatrix laboratories practices two levels of COC, the first being external COC and internal COC. The degree of custody tracking and documentation is driven by the final deposition of the laboratory data. Generally, if samples and their analytical results are subject to involvement as physical evidence or in a legal procedure; a full series of custody procedures must be implement. On the other hand, if samples or results are not subject to legal procedures, the COC process will end with the receipt of samples at the laboratory. A description of these two custody scenarios is presented as follows:

A. External COC

Samples only requiring external COC will have their custody tracked from sample collection to delivery at the laboratory. This process involves the completion of an TriMatrix external COC form, as presented in Figure 49. This form accompanies all sample containers prepared by TriMatrix to the sample collection site. Any sample or submittal received at the laboratory without an TriMatrix external COC form will initiate a process where the SC will complete the necessary external COC forms for carrier sign-off.

All external COC forms have a unique number for document control purposes.

B. Internal COC

Samples requiring strict COC will initiate the process by which all events or periods of sample handling will require a traceable document protocol.

The internal COC process involves the completion of an TriMatrix internal COC form for all phases of the analytical process. This includes

TriMatrix
Laboratories, Inc.

5555 Glenwood Hills Parkway SE • PO Box 888692 • Grand Rapids, MI 49588-8692

Chain of Custody Record

COC No.
№ 27056

Project Manager	Project Name		
Project No.	Sampler (Print)		
	Sampler Signature		

No. of Containers	No's Correspond to Bottle Packing List		For Lab Use Only
			Rack/Tray No:
			Lab Project #

Date Sampled	Time Sampled	Matrix*	Composite	Grab	Sample Identification	No. of Containers	Container Type	Analysis Required/Comments	Sample No.	Filtered Date/Time
							1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20			
							1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20			
							1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20			
							1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20			
							1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20			
							1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20			
							1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20			
							1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20			
							1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20			

Relinquished By:	Date/Time	Received By:	Received to Lab By:	Date/Time	Logged in By:	Date/Time

* Matrix: Water (WTR), Wastewater (WW), Soil (SOIL), Sludge (SLG), Air, Oil, Waste (WASTE)

Figure 49

sample extractions distillations or digestion's, sample analyses and final sample disposal. An example of the TriMatrix internal COC form is presented in Figure 50.

All internal COC forms are maintained in a series of submittal or delivery group folders. These folders are held within the secure confines of the laboratory and or locked project file storage areas.

C. Sample Security

All samples whether under external or internal COC protocols are maintained in a limited access secured area. This level of security is applied to all phases of the analytical process from sample log-in to final sample disposal.

D. Sample Disposal

All samples received at the TriMatrix laboratories are subject to some type of disposal activities upon completion of the analytical process. The general categories or procedures for sample disposal are: 1) samples are returned to the client for disposal, 2) samples are classified as non-hazardous and disposed via the local WWTP and municipal landfill or 3) samples are deemed hazardous and require proper hazardous waste disposal protocols.

The procedures detailing these three disposal activities are detailed in the TriMatrix "Waste Disposal SOP for Analytical Samples".

17-J[redacted]96 TRIMATRIX LA[redacted]ORIES, INC. - GRR [redacted] 115

CHAIN OF CUSTODY REP[redacted] FOR AN ENTIRE SUBMITTAL

CLIENT: EARTH TECH-Grand Rapids Office PROJECT: Analytical Services

SUBMITTAL: January 16, 1996 Samples PROJECT: 33086-1

Parameter: IRON, TOTAL Method: ICP/EPA/WT Ref Cit: EPA-200.7/6010A Matrix: WATER

SAMPLE #	REMOVED BY: (SIGNATURE)	DATE & TIME REMOVED	RELINQUISHED BY: DATE & TIME	RECEIVED BY: DATE & TIME	DATE & TIME RETURNED
136055	David Johnson	1-17 96 8:00A			1-17-96 12:00 PM
136056	DWS				
136057	DWS				
136058	DWS				

Figure 50

4.3 CALIBRATION AND CALIBRATION VERIFICATION

This section describes procedures for maintaining the accuracy of all the instruments and measuring equipment which are used for conducting laboratory analyses. These instruments and equipment should be calibrated prior to each use or on a scheduled periodic basis.

Calibration of laboratory instruments, conventional wet-chemistry methods, and equipment is performed to verify that the analysis portion of the total testing process is functioning properly, in range, at the required sensitivity. A separate written calibration procedure and calibration record is maintained for each laboratory instrument and applicable equipment. The procedure will specify calibration frequency, stability, and calibration steps based on analytical method requirements and instrument or equipment manufacturers recommended schedule of calibration.

Initial calibration is performed using standards of certified value to establish the linear range of the analysis for those concentrations of interest. Calibration verification is done immediately after initial calibration and on a continuing basis during an analysis batch. A standard from a different vendor or stock, than the initial calibration is used for calibration verification, in most cases.

Calibration activities are divided into four categories:

Field Equipment
Laboratory Instruments
Classical Inorganic (wet-chemistry) Methods
Secondary Calibration Group, including laboratory equipment requiring calibration (such as ovens, balances, thermometers).

4.3.1 Field Equipment

Perform daily calibration checks on field equipment as described in 4.3 before any field operations are performed with the equipment. Follow the written calibration procedure for each individual piece of field equipment. Tag any equipment that did not pass daily requirements as out-of-service in accordance with the standard operating procedure. The equipment is held out of service until repairs and successful recalibration occurs. A summary table of all calibration procedures and frequencies is included (Table 6).

TABLE 6
FIELD EQUIPMENT CALIBRATION

Equipment	Method Reference	# Standards Initial Calibration	Type of Curve	Frequency of Calibration	Acceptance/ Rejection Criteria Initial Calibration	Frequency of Continuing Calibration Verification	Acceptance/ Rejection Criteria Continuing Calibration Verification
Field Gas Chromatograph	USEPA SW846 Field Manual;	5	Linearity 0.995 corr. coef.	As Needed	20% D	Every 10 Samples	Same criteria as initial calibration
Conductivity Meter	*	2	---	Initial	± 5% of Value	Daily	---
Dissolved Oxyge Meter		---	---	Initial	± 5% of Value	Daily	---
Temperature Probes		---	---	Initial	± 5% of Value	Daily	---
pH Meter		3	Linearity	Initial	>0.995 correlation coefficient	Daily	---
Turbidity Meter		1 Std. each range Low 1.0 NTU Mid 10 NTU High 100 NTU	---	Initial	Instrument manually set at 100% reading for standard	(1 std in conc. range) every 10 samples each range	± 10% of Value
Salinity Meter		1	---	Initial	± 5% of Value	Daily	---

*USEPA Region Engineering Support Branch, Standard Operating Procedures and Quality Assurance Manual; USEPA Compendium of Superfund Field Operations Methods.

4.3.2 Laboratory Instrumentation

Calibration of laboratory instruments is based on approved written procedures. Records of calibration, repairs, or replacement are filed and maintained by the designated laboratory analyst. These records are filed at the location where the work is performed and are subject to QA audit. For all instruments, the laboratory maintains in-house spare parts or service contracts with vendors. A summary table of all calibration procedures and frequencies is included (Table 7). Tag any instrument that does not pass daily requirements as out of service per the written procedure. Hold the instrument out of service until repair or successful recalibration occurs.

4.3.2.1 Inorganic/Classical Chemistries

Inorganics analysis utilizes a wide variety of wet-chemical procedures and instruments. Calibration steps may vary depending on the specific analytical method being utilized. However, certain general principles of calibration apply to all inorganics testing. Every method must be calibrated before an analysis is performed. Using a group of certified standards, the linear range is defined. The calibration is checked on a continuing basis to be certain that the method is within the required test parameters. Any failure of a continuing calibration check will require that a recalibration be performed and any samples analyzed since the last acceptable calibration be reanalyzed.

All inorganics calibrations must meet the specific requirements described below unless the method or equipment necessitate some modifications.

The instrumentation used to conduct these analyses is calibrated by use of a minimum of five calibration standards prepared by dilution of stock solutions. One standard is prepared at the detection limit of the analyte of interest while the other standards bracket the concentration range of the samples.

A laboratory control standard, prepared from a different stock solution than that used for preparation of the calibration standards, is prepared and analyzed with each batch of 20 samples. An initial calibration blank and initial calibration verification are analyzed at the beginning of each run. A continuing calibration standard and continuing calibration blank will be analyzed after each batch of 10 samples. The value of the continuing calibration standard concentration must agree within ± 15 percent of the initial value or the appropriate corrective action is taken which may include recalibrating the instrument and reanalyzing the previous 10 samples.

4.3.2.2 Atomic Absorption/Emission Systems

The atomic absorption spectrophotometer (AAS) and inductively coupled plasma emission spectrophotometer (ICP) instruments are calibrated by use of a minimum of three calibration standards (1 calibration standard for ICP) prepared by dilution of certified stock solutions. One standard is prepared at the detection limit of the analyte of interest while the other standards bracket the concentration range of the samples. Calibration standards contain acids at the same concentration as the digestates. A laboratory control standard, prepared from a different stock solution than that used for preparation of the calibration standards, is prepared and analyzed after each ten samples or each two hours of continuous operation. A continuing calibration standard is analyzed after each batch of 10 samples. The value of the continuing calibration standard concentration must agree within method specifications (generally 5-10 percent) of the initial value or the appropriate corrective action is taken which may include recalibrating the instrument and reanalyzing the previous ten samples.

4.3.2.3 Gas/Liquid Chromatography

Analysis done by gas chromatography follows USEPA protocols. The instrument is calibrated using five point calibration curves for volatile compounds and semi-volatile compounds. Continuing calibrations are performed after every ten samples. The value of the continuing calibration standard must agree within ± 15 percent of the initial value or the appropriate corrective action is taken, which may include recalibrating the instrument and reanalyzing the previous ten samples.

4.3.2.4 Mass Spectroscopy

Prior to calibration, the instrument(s) used for gas chromatograph/mass spectrometer (GC/MS) analyses are tuned by analysis of p-bromofluorobenzene (BFB) for volatile analyses and decafluorotriphenyl phosphine (DFTPP) for semi-volatile analyses. Once the tuning criteria for these reference compounds are met, the instrument is initially calibrated by using a five point calibration curve. The instrument tune will be verified each 12 or 24 hours of operation (depending on method requirements). Continuing calibration is verified as specified in the method. The calibration standards are commercially available certified standards and are spiked with internal standards and surrogate compounds.

TABLE 7
INSTRUMENT CALIBRATION

Instrument	Method Reference	# Standards Initial Calibration	Acceptance/Rejection Criteria Initial Calibration	Frequency of Calibration	Frequency of Initial Calibration Verification	Acceptance/ Rejection Criteria Initial Calibration Verification	Frequency of Continuing Calibration Verification	Acceptance/ Rejection Criteria Continuing Calibration Verification
Flame AA	SW-846	4	Correlation coefficient must be ≥0.995	At least daily, or as required (when CCV fails acceptance criteria).	Every calibration	90-110%R	Every 10 samples	90-110%R
Cold Vapor AA	SW-846	4	Correlation coefficient must be ≥0.995			80-120%R		80-120%R
ICP	SW-846	1	Not Applicable			95-110%R		90-110%R
Graphite Furnace AA	SW-846	4	Correlation coefficient must be ≥0.995			90-110%		90-110%
pH Meter	SW-846	2	± 0.1 STD units of true value	Every batch	Every calibration	± 0.1 STD units of true value	Every 10 samples	± 0.1 STD unit of true value

TABLE 7
INSTRUMENT CALIBRATION

Instrument	Method Reference	#Standards Initial Calibration	Acceptance/Rejection Criteria Initial Calibration	Frequency of Calibration	Frequency of Initial Calibration Verification	Acceptance/ Rejection Criteria Initial Calibration Verification	Frequency of Continuing Calibration Verification	Acceptance/ Rejection Criteria Continuing Calibration Verification
DL-12 Autotitrator	SW-846	2	± 0.1 STD units of true value	Every batch	Every calibration	85-115%	Every 10 samples	85-115%
Lachat: SO4 Cl	EPA 600/4-79-020 Method 375.4 Method 325.2	 6 7	 Nonlinear Nonlinear	Every batch	Every calibration	85-115%	Every 10 samples	85-115%
Phenol (Total)	SW846-9066	4-6	≥0.995					
Cyanide (Total)	SW846-9012	6	≥0.995					
Cyanide (Amenable)			≥0.955					
TOC Analyzer-TOC	EPA-415.1	1	90-110%	Every Batch	Every Batch	85-115%	Every 10 samples	85-115%
GC-PID/ ELCD	SW-846 8010 SW-846 8020 SW-846 8021	5	RF <20% RSD or cal. curve	As needed, when CCV >15%D	As needed, with prep of new std.	15%D	Daily 10%, and at end of each batch	15%D

TABLE 7
INSTRUMENT CALIBRATION

Instrument	Method Reference	# Standards Initial Calibration	Acceptance/Rejection Criteria Initial Calibration	Frequency of Calibration	Frequency of Initial Calibration Verification	Acceptance/ Rejection Criteria Initial Calibration Verification	Frequency of Continuing Calibration Verification	Acceptance/ Rejection Criteria Continuing Calibration Verification
GC-ECD	SW-846 8080 SW-846 8150	5	Linearity <20% RSD	As needed when CCV >15%D	As needed with each new std. Quarterly at a minimum	15%D	Every 10 injections	Primary column %D <15. Conf column %D <20. Breakdown criteria: DDT <20% Endrin <20%
GC/MS-volatiles	SW-846 (8240, 8260)	5	%RSD <35% (CCC) 1,1-dichloroethene; chloroform 1,2-dichloropropane; toluene ethyl benzene; vinyl chloride RF> 0.30 (SPCC) chloromethane; 1,1-dichloroethane; bromoform (0.25); 1,1,2,2-tetrachloroethene; chlorobenzene	As needed	As needed	20%D	Every 12 hrs.- (SW-846) Ever 24 hrs.- (USEPA 600 Series)	CCC %D <25% same SPCC criteria as initial calibration

TABLE 7
INSTRUMENT CALIBRATION

Instrument	Method Reference	# Standards Initial Calibration	Acceptance/Rejection Criteria Initial Calibration	Frequency of Calibration	Frequency of Initial Calibration Verification	Acceptance/ Rejection Criteria Initial Calibration Verification	Frequency of Continuing Calibration Verification	Acceptance/ Rejection Criteria Continuing Calibration Verification
GC/MS-semi-volatiles	SW846-8270	5	% RSD <30% (CCC) acenaphthene 1,4-dichlorobenzene hexachlorobutadiene N-nitroso-diphenylamine di-octylphthalate fluoranthene benzo(a)pyrene 4-chloro-3-methylphenol 2,4-dichlorophenol 2-nitrophenol phenol pentachlorophenol 2,4,6-trichlorophenol RF>0.05(SPCC) N-nitrosodipropylamine hexachlorocyclopentadiene 2,4-dinitrophenol 4-nitrophenol	As needed	As needed	20%D	Every 12 hours	CCC %D <30%. Same SPCC criteria as initial cal.

4.3.3 Laboratory Equipment

Perform calibration at the intervals described below on equipment such as thermometers, ovens, balances, furnaces which are stable and retain their calibration over a long period of time.

Balances

Perform a full calibration annually by using an outside contractor who is certified. Use class S or higher weights that are traceable to the National Institutes of Standards and Technology (NIST). Weights must be recertified every three years. Calibrate balances to approximately 95 percent of capacity. Use testing intervals of approximately 5, 10, 15, 20, 25, 30, 40, 50, 60, 70, 80, 90, and 95 percent of capacity. Acceptable calibration readings for direct reading balances are plus and minus the combined tolerance of the calibration weights and the balance reproducibility, expressed as total tolerance in grams.

Perform a check of the balances each work day using certified calibration weights to cover each weighing range. The acceptable calibration readings are the same as those described above for annual calibration.

Maintain written records of annual calibrations and daily calibration checks. Place a sticker on the balance showing the date of the current calibration, name of person performing the calibration, and the next due date.

Thermometers

Use a primary NIST reference thermometer to check at least two points in the temperature range of working thermometers. Perform annually for all mercury thermometers and quarterly for all dial type thermometers.

If equipment fails calibration, remove the item from service and tag it to prevent use. Personnel performing the calibration should be also alert for any condition which renders a piece of equipment inoperable or unfit for use; for example, inspect thermometers to ensure that mercury or alcohol columns are not separated and that no other damage is present. If an equipment malfunction is noted during

calibration, the equipment is to be tagged and removed from service. The equipment is held out of service until repairs and successful recalibration occur. Record all malfunctions, repairs, and recalibrations in the appropriate instrument maintenance and calibration logs.

Maintain records for each piece of equipment requiring calibration, showing equipment description and identification number, calibration frequency and acceptable tolerances, the specific calibration procedure used, personnel performance calibration, date, reference material used, calibration results including acceptance or failure, removal from service, repairs, and date and authorization for return to service.

4.4 DATA REDUCTION, VALIDATION, AND REPORTING

All analytical data generated by TriMatrix laboratories is thoroughly reviewed. Data verification begins with the analyst, because it is the basic responsibility of the analyst that the data is complete and correct and that all applicable methods and standard operating procedures have been followed. If results are not acceptable, it is the duty of the analyst to perform the appropriate corrective action and to thoroughly document that action.

Data reduction is the process by which raw analytical data is tabulated and calculated. Data validation is the review of the data generation and reduction process according to an established set of guidelines. This verification of data generation and reduction steps, performed by the analyst, constitutes the first level review of the analytical data.

The data is then passed to the laboratory area supervisor or designated validator who performs the second level review of the test results. This independent review is also conducted according to an established set of validation guidelines.

The data is then entered and verified (via double blind entry) into the laboratory LIMS system. Upon completion of the verification process, the data accompanied by a printed report is returned to the area supervisor r designated validator for final approval. This process constitutes the third level of review.

The data reporting format, which is generated by the client services group, will vary depending upon the client project requirements. A wide variety of reporting packages are available, including customized presentations. Also, a fourth level review is performed at the point of report generation by client services.

The project chemist subsequently performs a fifth level review to ensure that the data fulfills the total objectives of the customer and is presented in an understandable manner. Further, the Quality Assurance Supervisor performs a random audit of approximately 5% of all laboratory data before the information is reported to the client (Refer to Figure 51).

4.4.1 Field Data

All data reduction, validation, and reporting for field activities must meet the same requirements as those listed in section 4.4.2 below involving laboratory data. In addition, field testing places an added responsibility on the analyst because it is much more difficult to return to the site for a retest at a later time than for the laboratory analyst who usually has sufficient sample to perform a requested retest in the laboratory. Furthermore, many of the field instruments, such as those measuring pH, dissolved oxygen, turbidity, temperature, and specific conductance, require a manual data printout from a computer interface. The analyst is responsible for immediate tabulation and calculation of raw data in the field. The field section supervisor must perform a prompt, on-site validation of field data before the opportunity is lost to perform any necessary field retests. Where possible, data which has been validated should be entered into a field electronic database using a portable (laptop) computer; this can facilitate transmission via modem to the laboratory and also the prompt transfer of data by diskette.

4.4.2 Laboratory Data

Data Reduction

Analytical data from most of the instruments at the TriMatrix laboratory is generated through computer programs interfaced to the instruments. For example, GC/MS data is processed through a local PC utilizing the manufactuer's software. Gas chromatographs are interfaced to turbochrome software from PE Nelson.

Computer data reduction utilizes software that has been validated for its intended purpose. The computer program usually incorporates all calculations, dilutions, etc. eliminating the need for further manual data reduction. Where manual data reduction is required, it is performed according to the written standard operating procedure for that analysis.

Data Reduction, Validation and Reporting

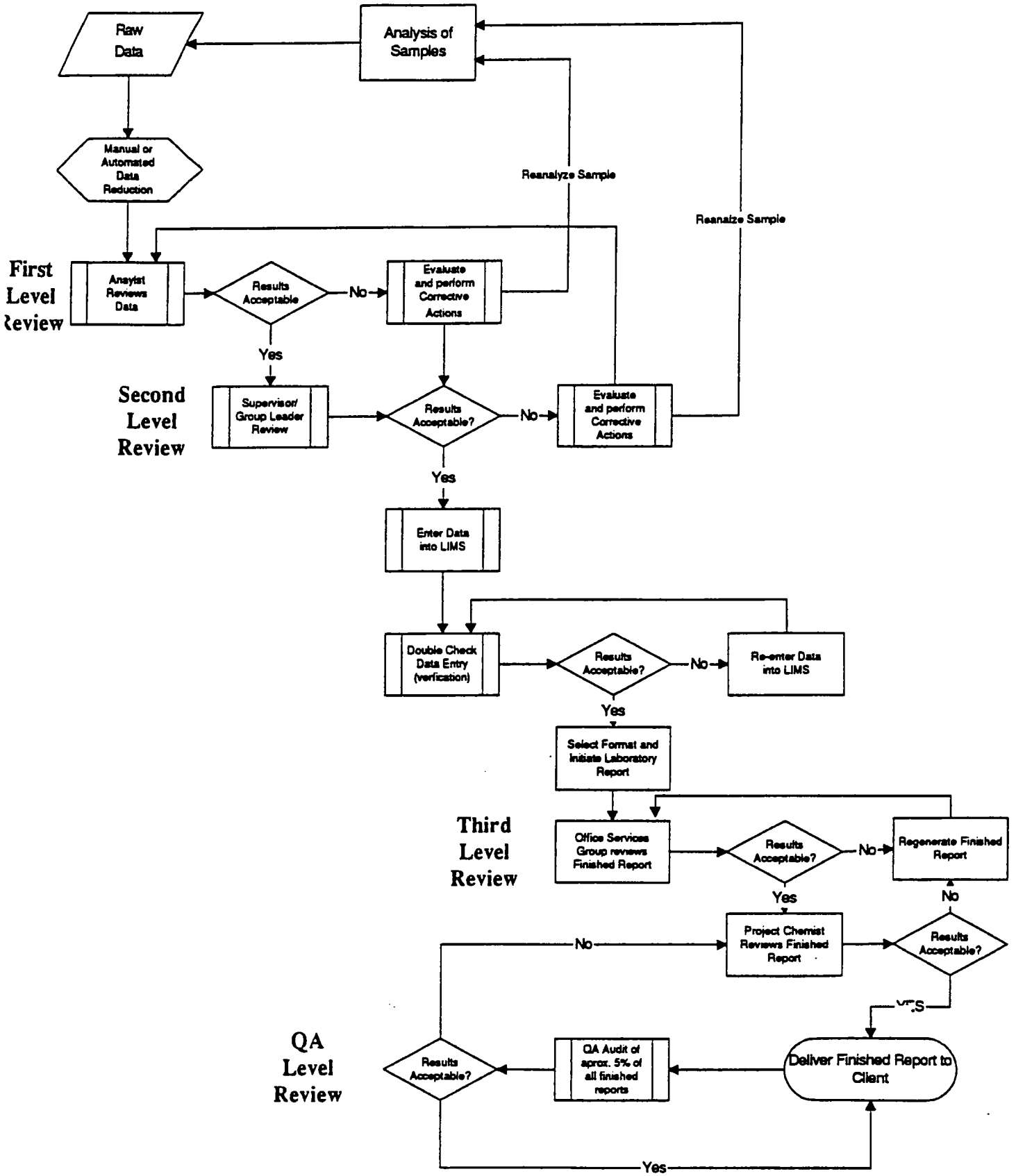


Figure 51

Data Validation

First Level Review: Raw Data

The laboratory analyst follows a written data validation check list to ensure:

Proper analytical sequence sample preparation information is correct.

Applicable standard operating procedures were followed.

Calibration has been performed properly.

Analytical results are complete.

Holding times have been met.

Analysis information is correct.

Calculations are performed properly.

Method criteria are met (sound analytically).

Quality control samples are within established limits.

Any special sample preparation or analytical requirements have been achieved.

Documentation is complete.

Raw data

Chromatograms, instrument printouts

Worksheets

Case narrative or qualifier pages

All analytical abnormalities have been noted.

Corrective actions are thoroughly described.

Good recordkeeping practices have been followed.

Any problems are communicated to supervisor.

Second Level Review: Raw Data

The laboratory area supervisor or designated validator ensures that:
quality control samples are within established limits
qualitative and quantitative results are correct
calibration data is correct per the analytical method
the analytical method has been followed
documentation is complete and data is legally defensible
the laboratory data package is ready for data entry and verification
any problems are communicated to analyst and, when appropriate for wider review, to the quality assurance supervisor.

Third Level Review: Raw Data and Finished Client Report

The client services staff ensures that data is accurately presented in final report.

Fourth Level Review: Finished Client Report

The project chemist ensures that:
Requirements of the client have been met.
The overall presentation of data to the client is in an attractive, understandable format.

Quality Assurance Review (Fifth Level of Review) Finished Client Report

The QA supervisor randomly audits approximately 5% of all data generated to ensure that:
Good record-keeping practices have been followed.
Quantitative results are correct.
Data is legally defensible.
Holding times have been met.
Calibration checks are sufficient.
QC results are acceptable.
Documentation is complete.
Corrective actions have been thoroughly described.

"Common Sense" Guidelines For All Levels of Review

In addition to the formal data validation guidelines listed above for the analyst, supervisor, project chemist, and QA supervisor , there are many practical questions that all of these persons need to keep in mind when reviewing data and finished client reports. Among these "common-sense" evaluations of laboratory data are the following important considerations:

Data makes good, sound, practical sense.

Multiple runs of the same samples relate, match, or are within acceptable range.

Data from complimentary analyses compares - COD>BOD>CBOD

Total cyanide ≥ amenable and free cyanide

Sum of anions and cations should not exceed specific conductance.

Total solids ≥ suspended and dissolved solids.

TKN ≥ organic N + ammonia N

Inorganic N = ammonia N + nitrate N + nitrite N

TOC < BOD or COD

Total phosphorus ≥ orthophosphorus

(Conductivity) x 0.6 - 0.9 = total dissolved solids

TPH-GRO > BTEX

Analytical run looks good; proper decisions were made.

Peaks from chromatogram or instrument printout look normal.

Computer identifications are correct.

Are hits real, especially low level?

Know and be sensitive to common laboratory contaminants.

Know area/analytical method pitfalls-be extra cautious.

All practices are sound and are supported by documentation-no appearance of random decisions.

Data Reporting

When laboratory analyses and review of data are completed, laboratory reports for clients are generated by the office services group according to a written standard operating procedure. Each step of the procedure is critical to the prompt generation of an accurate and complete laboratory report by office services personnel.

Data entry requires a computer password and is restricted to certain individuals on the office services staff. An identification number for the entry person tracks each data item. Restricted entry to the computer database protects the client data. Data entry is verified for correctness by a second person after the initial entry.

To initiate a laboratory report, the office services staff must review the project for special instructions and completion of data entry before accepting the project as complete. The project is subsequently assigned a completion date and removed from the daily laboratory work schedule.

A format is selected from among many possibilities (regular, TCLP, state agency, BNA, VOA, Pest/PCB, etc.). If qualifiers or case narrative are involved, then the appropriate entries are made from a standardized list using uniform wording.

When the laboratory report content is complete, a printout is reviewed for accuracy and appearance before a final version is forwarded to the project chemist. A fourth level review by the project chemist is the last step before the finished laboratory report is delivered to the client.

4.5 VERIFICATION PRACTICES - EXTERNAL/INTERNAL QUALITY CONTROL

4.5.1 Standard Reference Materials

A crucial step in the generation of quality data is the purity and traceability of reference materials used in the analyses. Reference materials may be physical standards (such as certified thermometers and weights used to calibrate laboratory thermometers and balances) or chemical standards (used to establish and check operational calibration of analytical methods). Physical standards should be traceable to the National Institute of Standards and Technology (NIST). Physical standards must be recalibrated every two years by an external vendor who is certified to perform the calibration. Chemical reference materials of high quality can usually be obtained from reliable commercial vendors. In addition, Standard Reference Materials (SRM's) are available from NIST for a significant number of analytes and various matrices. For a given analysis, standard reference materials should be kept on hand from more than one vendor source. During the testing operation standard reference materials from different vendor sources should be cross-checked with each other. That is, a calibration curve would be performed using standards from a different vendor source.

4.5.2 Internal Quality Control Programs

4.5.2.1 TriMatrix Analytical Services routinely adds samples to the sample stream to demonstrate that the total testing process is operating within prescribed limits for accuracy and precision. With the exception of Blanks, the concentration of these quality control samples is known prior to the analysis. Types of Quality Control Samples are in Table 8.

TABLE 8

Blank Type	Abbreviation	Description	Frequency of Use
Method Preparation Blank	MPB	This blank has been carried through the entire analytical process including any pretreatment procedures. The MPB will monitor any contaminants that may affect the sample results. General acceptance limits for the MPB are ± the test reporting Limit. If contamination is detected in the MPB, all samples with analyte concentratins <5X the test reporting limit, must be flagged for re-extraction or digestion. If it is not possible to re-prep the samples, then all analyses for that batch must be qualified.	One per analytical batch
Daily Instrument Analytical Blank	BLK	Analyzed once per day and/or at the beginning of analytical operations, this blank is used to detect any contamination in the instrument system.	One per day or per analytical batch

TABLE 8

Blank Type	Abbreviation	Description	Frequency of Use
Initial Calibration Blank	ICB	This is reagent blank that is analyzed as a sample after a calibration curve has been generated for an analysis. The ICB will check how close the curve passes through zero. Acceptance limits for an ICB are ± the Test Reporting Limit. If the ICB is outside these limits, the instrument must be recalibrated and the ICB reanalyzed.	One per analytical batch or as specified in the analytical method.

TABLE 8

Blank Type	Abbreviation	Description	Frequency of Use
Continuing Calibration Blank	CCB	The continuing calibration blank is a reagent blank that is analyzed as a sample, generally after 10 samples have been tested. The CCB must be run prior to re-zeroing an instrument, unless this practice was performed for each previous sample. The CCB will verify whether significant instrument draft has occurred during the analytical run near the test method detection limit. General acceptance limits are ± the test reporting limit. If the CCB falls outside the acceptance limits, the instrument must be recalibrated and the previous 10 samples reanalyzed. For automated tests where run data is generated after all analyses are completed, 10 samples before and after the unacceptable CCB must be reanalyzed, i.e., all sample results must be encased in acceptable CCB. The reanalysis must also include the ICB and ICV QC samples.	Every ten samples/or as specified in the analytical method.

TABLE 8

Blank Type	Abbreviation	Description	Frequency of Use
Field Trip Blank	FTB	These are used with VOA vials where there is the possibility that organic contaminants may diffuse through the teflon-faced silicone rubber septum of the sample vial. A field trip blank vial filled with organic-free water accompanies the sample containers to and from a client location, at the discretion of the client, may be analyzed along with the samples.	One per sample shipping container
Storage Blank	STB	Reagent-grade water (40 ml aliquot) is stored with samples in a client set. Per the discretion of the client, it may be analyzed after all samples in that set are analyzed. The purpose is to determine the level of contamination acquired during storage.	One per sample storage refrigerator or client sample set (if required)

TABLE 8

Blank Type	Abbreviation	Description	Frequency of Use
Sulfur Blank	SVB	This is a modified method blank that is prepared only when some of the samples in a batch are subjected to sulfur cleanup. It is used to determine the level of contamination associated with the sulfur cleanup procedure. When all of the samples are subjected to sulfur cleanup, then the method preparation blank serves this purpose. When none of the samples are subjected to sulfur cleanup, no sulfur blank is required.	When some of the samples in a batch undergo sulfur cleanup.

TABLE 8

CONTROL SAMPLES

Control Type	Abbreviation	Description	Frequency of Use
Laboratory Fortified Blank	LFB	This is a fortified method preparation blank in which an aliquot of deionized water has been spiked with a known amount of a stock reference standard or spiking solution. A blank spike is required for each digestion or distillation batch. The purpose of the blank spike is to verify the analyst's spiking procedure and assure that any matrix interference shown by the spike and spike duplicate is really matrix induced.	One per analytical batch

TABLE 8

CONTROL SAMPLES

Control Type	Abbreviation	Description	Frequency of Use
Laboratory Control Sample	LCS	The laboratory control sample is a reference sample of know value traceable to a reliable commercial vendor such as APG, ERA; or NIST or EPA. This sample may also be prepared in the laboratory from an independently prepared stock standard. The purpose of the LCS is to validate the accuracy of the analytical procedure. A BLK is usually analyzed prior to the analysis of the LCS. Acceptance limits for this QC type are based on a 95% confidence limit generated from historical data for this test. Also, a particular test method may have published acceptance limits for the LCS. If the LCS falls outside the established limits, the analytical batch must be flagged for re-extraction, re-digestion, or reanalysis. It is impossible to repeat the analysis (e.g. BOD test) then all data for the batch must be qualified.	One per analytical batch or per new calibration curve (organic analyses)

TABLE 8

Control Type	Abbreviation	Description	Frequency of Use
Initial Calibration Verification	ICV	The initial calibration standard is a mid-range standard. Generally one of calibration from the same stock is used. This standard is analyzed as a sample and compared with the standard curve. The ICV checks the precision of the curve. Acceptance limits for this standard are ± 10%, or as stated in a particular method. An ICB must be analyzed prior to testing the ICV. If the ICV falls outside the acceptance limits, the instrument must be recalibration and the process repeated.	Once per analytical batch or as specified in the analytical method

TABLE 8

Control Type	Abbreviation	Description	Frequency of Use
Continuing Calibration Verification	CCV	The continuing calibration verification is generally the same midrange standard that was analyzed as the ICV. The standard is analyzed as a sample and compared with the standard curve. The ICV will reveal any significant draft of the curve. Acceptance limits for this QC type are ± 10%, or as stated in a particular method. If the CCV falls outside the acceptance window, the instrument must be recalibrated and the previous 10 samples reanalyzed. For automated tests where run data is generated after all analysis is complete, 10 samples before and after the unacceptable CCV must be reanalyzed, i.e. all samples must be encased in acceptable CCV. The reanalysis must include the ICB and ICV QC types.	Every 10 samples

TABLE 8

Control Type	Abbreviation	Description	Frequency of Use
Contract Required	CRDL	A standard which contains the minimum level of detection acceptable under a contract Statement of Work must be analyzed for particular contract sample sets to demonstrate that detection limit can be met.	One per analytical batch for certain contract sample sets and methods only.
MATRIX QC SAMPLES			
Sample Matrix Spike	SPK	The sample matrix spike is an aliquot of a sample that has been spiked with a known amount of a stock reference standard or spiking solution. A the purpose of the SPK is to monitor sample matrix effects on the test. Acceptance limits for this QC type are based on the 95% confidence limits established for a test and matrix.	Every 10 samples for each matrix type, or as specified in the analytical method

TABLE 8

Matrix QC Type	Abbreviation	Description	Frequency of Use
Matrix Spike Duplicate	MSD	A matrix spike duplicate is an aliquot of the same sample used for the matrix spike (SPK). A spike duplicate is required for each matrix type within a digestion or distillation batch. A spike duplicate analysis may be required on an non-distilled or non-digested sample if the spike has indicated a matrix interference. The purpose of this duplicate spike is to confirm any matrix effects on the test. Acceptance limits for this QC type are based on the 95% confidence limits established for a test and matrix.	Every 10 samples for each matrix type or as specified in the analytical method
Sample Duplicate	DUP	The sample duplicate is a replicate analysis of a particular sample that has been analyzed previously during the sample analytical batch. The purpose of the duplicate is to monitor precision within the analytical process.	Every 10 samples for each matrix type

TABLE 8

Matrix QC Type	Abbreviation	Description	Frequency of Use
Field Duplicate	FDUP	This may be required to evaluate the uniformity of samples and sampling techniques at a field location. Acceptance limits for this QC type are based on established confidence limits, with generally two levels or ranges. The first range extends from the test reporting limit test to 10x the test reporting limit. The second range encompasses any values higher than 10x the MDL.	As required on a project basis
Post Digestion Spike	PDS	The post-digestion spike may be required, on a project basis, when a matrix precludes the use of pre-digestion spike.	One per analytical batch when required by project

TABLE 8

MISCELLANEOUS QC SAMPLES

	Abbreviation	Description	Frequency of Use
Surrogate Spike	SUR	For semi-volatile, volatiles, pesticide, PCB analyses, these surrogate compounds are added to every blank, sample, matrix spike, matrix spike duplicate, and standard. Surrogate compounds, which are used to measure analytical efficiency by measuring recovery, are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental samples.	Every QC and per batch for semi-volatile, volatile, pesticide, PCB analysis
Internal Standard	INS	These are compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for volatiles), at a known concentration, prior to analysis. Internal standards are used as the basis of quantitation of the target compounds.	Every QC and client sample per batch for volatiles and semi-volatiles

4.5.2.2 Presented in Table 9, is an example of a sequence of analysis using Internal Quality Control Samples for trace metals.
(Note that sequences may vary depending on the analytical method and the instrument being used).

TABLE 9
Sequence of Analysis
Metals Analysis - ICP

ID/WT Sheet Setup for Instrument Calibration and Verification

Sequence Number	Autosampler Position	Sample ID	Solution Name
1	1	is_init	BLANK
2	2	Std OPT A	Std OPT A
3	3	Std OPT B	Std OPT B
4	4	Std OPT C	Std OPT C
5	1	blank	BLANK
6	2	ICS-A	Std OPT A
7	3	ICS-B	Std OPT B
8	4	ICS-C	Std OPT C
9	5	ICV-A	ICV/CCV OPT A
10	6	ICV-B	ICV/CCV OPT B
11	7	ICV-C	ICV/CCV OPT C
12	1	Blk	BLANK
13	9	IEC-1	IEC-1
14	10	IEC-2	IEC-2
15	11	ICAP-7	Independent check
16	12	IV-19	Independent check
17	13	era1027	Independent check
18	14	Sn 10.0	Independent check
19	15	era3.11	Independent check
20	16		Sample1
21	17		Sample 2
22	5		CCV OPTA
23	6		CCV OPTB
24	7		CCV OPTC
25	18		CCB
26	19		Sample 4
27	20		Sample 5
28	21		Sample 6
29	22		Sample 8
30	23		Sample 9
31	24		Sample 10

4.5.3 External Quality Control Samples-Proficiency Testing

TriMatrix laboratories receive performance evaluation (PE) samples on a scheduled basis from state and federal regulatory agencies as well as certain client organizations. A summary of these PE samples is given below:

Evaluation Sample Identification	*Sample Type*	*Source*	*Frequency of Receipt of Samples*
WS	Drinking Water Certification	USEPA	Twice Per Year
WP	Waste Water (NPDES)	USEPA	Twice Per Year
DMR-QA	Discharge Monitor Reporting	USEPA	Once Per Year
PE	Environmental	Certain State Certifying Agencies	Once Per Year
PE	Environmental	Specific Clients	Once Per Year
Double-Blind PE	Environmental	Analytical Standards Inc. (ASI)	Once Per Year

TriMatrix laboratories receive written reports from sponsoring agencies grading not only the laboratory performance but also showing the comparison to other laboratories participating in the same performance evaluation studies. This provides feedback to laboratory personnel regarding the satisfactory use of analytical methods and equipment.

4.6 DATA ASSESSMENT PROCEDURES

4.6.1 Precision

Precision of laboratory analyses will be assessed by comparing the analytical results between matrix spike/matrix spike duplicate (MS/MSD) for organic analyses, and laboratory duplicate or MSD analyses for inorganic analyses. The relative percent difference (% RPD) will be calculated for each pair of duplicate analyses using Equation 4.6.1.

Equation 4.6.1

$$\% \text{ RPD} = \frac{S-D}{(S+D)/2} \times 100$$

Where:

S = first sample value (original of MS value)

D = second sample value (duplicate or MSD value)

4.6.2 Accuracy

Accuracy of laboratory results will be assessed for compliance with the established QC criteria using the analytical results of method blanks, reagent/preparation blank, matrix spike/matrix spike duplicate samples, equipment blank, and trip blanks. The percent recovery (%R) of matrix spikes will be calculated using Equation 4.6.2.

Equation 4.6.2

$$\% \text{ R} = \frac{A-B}{C} \times 100$$

Where:

A = the analyte concentration determined experimentally from the spiked sample;

B = the background level determined by a separate analysis of the unspiked sample, and

C = the amount of the spike added.

4.6.3 Completeness

The data completeness of laboratory analyses results will be assessed for *compliance with the amount of data required for decision making.* The completeness is calculated using Equation 4.6.3.

Equation 4.6.3

$$\text{Completeness} = \frac{\text{valid data obtained}}{\text{total data planned}} \times 100$$

4.7 PROCEDURES FOR CORRECTIVE ACTION

When an even occurs which is outside the normal operating parameters for the total testing process, a non-conforming event or process deviation has occurred which places the process out of control, and corrective action is required. A written standard operating procedure (plan for corrective action) provides the steps for dealing with an out-of-control testing situation. The assessment of whether the process is out of control is based on predetermined limits for laboratory operations. Nonconformances based on statistical analysis or quality control samples are readily apparent and easy to identify. A process deviation which does not have an directly observable impact on data quality is more difficult to discern. Examples of the latter, more subtle types of nonconformances include volatiles samples not properly stored; oily layers in certain types of samples which may interfere with the analysis; a water-soaked sample label whose information is barely readable; a cluttered and disorganized laboratory bench which increases the probability that a mix-up will occur; a person too preoccupied with interruptions or distractions to concentrate on a task; the expected quantity has not been removed from a sample container after the testing has commenced. Discovery of a nonconforming event or process deviation

can result from the observations of a staff member, a review of laboratory data at any level, or as the result of a quality audit.

The overall scheme of a correction action plan can be outlined as follows:

1. Define the problem.
2. Assign responsibility for evaluating the problem.
3. Determine thorough investigation of all the pertinent facts what the probable cause of the problem is.
4. Define a corrective action to eliminate the problem.
5. Assign responsibility for carrying out the corrective steps and implement the action.
6. Follow-up to ensure that the problem has been eliminated.

Specific responsibility for implementing corrective action is as follows.

It is the responsibility if the analyst or other employee who observes a non-conforming event to:

- Identify and define the problem.
- Write a non-conformance report (refer to Figure 52).
- Investigate and attempt to determine the cause of the problem.
- Report the problem promptly to the immediate supervisor along with a recommended course of action to eliminate the problem.
- Accept responsibility for implementing the corrective action approved by the supervisor.
- Implement the corrective action and evaluate its effectiveness.
- Verify that the corrective action has eliminated the problem.
- Document the corrective action in laboratory notebook or on worksheet.

It is the responsibility of the laboratory area supervisor to:

- Review the problem and the proposed corrective action.
- If the reporting person does not have a remedy, to work together with the person to determine a satisfactory solution.
- Assign the final corrective action steps to be performed.
- Sign and date corrective action entry in the laboratory notebook or on worksheet after completion.

It is the responsibility of the QA supervisor to:

- Review, sign, and categorize every nonconformance/report memo for later inclusion in the QA monthly report.
- Randomly review corrective action documentation in laboratory record through internal audits to ensure that adequate records are being kept.

In general, there are three major areas where corrective action is required, and these categories are described below.

4.7.1 Quality Control Failures

These are usually handled within the laboratory by the analyst.

Indications of Nonconformance

Blanks, laboratory controls, spikes, or surrogates contain contamination greater than acceptable levels.

Suspicious trends in spike recoveries or relative percent difference (RPD) between duplicates.

Initial instrument blank, initial calibration standards. QC check standards, continuing calibration standard spikes, or method blanks are outside acceptance criteria.

The method blank or instrument blank analysis exceeds the detection limit for that analyte.

Recommended Corrective Action

Prepare another instrument blank. If the response is still greater than the reporting limit, look for sources of contamination in reagents, laboratory working environment, instrument.

Reanalyze standard. If results are still unacceptable, prepare new standards. If necessary obtain new primary standards.

Reanalyze continuing calibration standard. If necessary, recalibrate and reanalyze samples since last successful continuing calibration.

Evaluate preparation of spikes, spiking techniques, spiking equipment and materials.

4.7.2 Procedural Failures

These are usually handled by the laboratory area supervisor and the quality assurance supervisor.

Indications of Nonconformance

There are unusual changes in detection limits.

Statistical quality control data (SQC) is demonstrating unacceptable trends or is outside the warning or acceptance limits.

Deficiencies are evidenced on performance evaluation samples or internal or external audits.

Clients express concern about the quality of their data.

Recommended Corrective Action (per USEPA guidelines)

Review the method with the analyst.

Reanalyze the samples and evaluate the results.

Recalibrate the instrument or analysis method with freshly prepared standards and reanalyze the samples.

Re-extract and reanalyze the samples per the method.

Evaluate the data and sample behavior and investigate any possible chemical interferences.

Re-run the samples using the method of standard additions.

Check the instrument for possible maintenance deficiencies.

Seek additional help from other analysts or provide additional training for personnel involved.

Perform a system audit to evaluate corrective action measures.

4.7.3 Test Specification Failures

These are usually handled by the analyst, laboratory area supervisor, and the quality assurance supervisor.

Indications of Nonconformance

Quality control check standard data is outside the acceptance limits defined for that analyte.

Recommended Corrective Action

Review the method with the analyst.

Reanalyze the check standard and evaluate the results.

Prepare fresh check standard or new primary standard.

Recalibrate the instrument or analysis method.

Switch to a different standard vendor.

Investigate possible chemical interferences.

Check the instrument for possible maintenance deficiencies.

Retrain the analyst.

4.7.4 Customer Complaints

The Quality Assurance Supervisor coordinates with the client services staff to receive quality feed back from clients. It is the responsibility of the QA Supervisor to communicate any customer complaints to the laboratory operating areas and to follow-up on corrective action taken to prevent a recurrence.

4.8 DEPARTURE FROM DOCUMENTED PROCEDURES

4.8.1 Management Policies

4.8.1.1 Any departure from a laboratory written standard operating procedure not directly involving sample analysis or processing must be approved by the immediate supervisor. The supervisor must file a Nonconformance Report.

4.8.1.2 Any departure from a USEPA or compendia analytical method involving sample processing or sample analysis must be justified in writing by they analyst and laboratory supervisor. The prior written approval of the laboratory director must be received before performing the analysis. Furthermore, the Laboratory Director must notify in writing the Vice-President, Laboratory Division, within 48 hours of this deviation. The laboratory director must also file a Nonconformance Report.

(Note: the exception to this requirement in 4.8.1.2 is those items in the analytical methods where a written justification for technical and scientific reasons has been determined by the analyst and approved by the Laboratory Director as a deviation from the analytical method).

4.8.2 Method Modification and Variances

Modification of and variances in analytical methods, except for the deviations justified in writing and approved per section 4.8.1.2 above, are strictly prohibited.

4.9 PERFORMANCE AND SYSTEM AUDITS

4.9.1 Internal Audits

4.9.1.1 System Audits

At least once each month, the Quality Assurance Supervisor or his designate performs an audit of at least one of the laboratory systems listed below, so that all laboratory systems receive a formal audit at three times per year.

A). Sample Handling and Control
B). Sample Analysis
C). Records Processing and Control
D). Support Systems (such as air handling, DI water, analytical balances, raw materials, etc.)

The system audits are to be used to determine that each component within a laboratory system is functioning properly and adheres to the appropriate standard operating procedures, analytical methods, and requirements of the Quality Assurance Manual. Develop formal checklists to use in System Audits to ensure that consistency of inquiring is maintained from month-to-month.

4.9.1.2 Documentation Audits

At least once each month, the Quality Assurance Supervisor or his designate performs an audit of the laboratory documentation (laboratory notebooks, benchsheets, instrument run logs, client file folders, etc) to assess the thoroughness and completeness of the documents. The guidelines for documentation (DO's and DO NOT's of GOOD RECORD KEEPING) are to be used as a benchmark in evaluation of documentation. Develop formal checklists for Documentation Audits to ensure that consistency of inquiry is maintained from month-to-month.

4.9.1.3 Surveillance Audits

At least once a month, the Quality Assurance or area Supervisor or their designate observes an analyst in detail as a test is being performed. Attention is given to general laboratory demeanor (orderliness, cleanliness, good laboratory practices in measuring, documentation, etc.) as well as to adherence to analytical methods and standard operating procedures., Develop formal checklists for surveillance audits to ensure that consistency of inquiry is maintained from month-to-month.

4.9.1.4 Quality Assurance Reports to Management

The Quality Assurance Supervisor issues a written report to the Laboratory Director within 1 week of an audit. The report should detail any deficiencies identified as well as recommended corrective action. The report should designate how follow-up on corrective actions by the Laboratory Area Supervisor and the Quality Assurance Supervisor will occur.

4.9.2 External Audits

4.9.2.1 On-Site Audits

A well-run laboratory should have nothing to fear from a visit by external auditors. Therefore, the audits of the laboratory conducted by regulating agencies and client organizations are to be perceived by the laboratory staff as positive occurrences-learning experiences and opportunities to hear suggestions from knowledgeable persons on how operations might be improved. Consequently, the laboratory staff is to be open and cooperative with external auditors. Formal follow-up using written summaries of external audits is to be carried out to ensure that any suggested improvements are thoroughly evaluated.

4.9.2.2 *Double-Blind Evaluation Studies*

TriMatrix Laboratories participate in at least one double-blind sample program each year. An example is the current arrangement with Analytical Services, Inc. (ASI) to provide such samples. In this program, only the Quality Assurance Supervisor is aware of the arrival of these proficiency check samples, since ASI uses one or more fictitious engineering companies to conduct the entire transaction with the laboratory. A follow-up report is issued by ASI to the Quality Assurance Supervisor, showing not only the laboratory performance but the ranking among the many other laboratories which participate in the double-blind evaluation study.

TriMatrix Laboratories, Inc.

ENVIRONMENTAL LABORATORY

NON-CONFORMANCE REPORT

Client Name:	_____		**Document Control No:**	_____
Project Description:	_____		**Submittal Number:**	_____
	_____		**Sample Number(s):**	_____
Lab Area/Activity:	Sample Receiving:	☐	Extraction Lab:	☐
	Sample Log-In	☐	Inorganic Lab:	☐
	Sample Storage:	☐	Metals Lab:	☐
	Client Services:	☐	Volatiles Lab:	☐
	Customer Compliant:	☐	Semi-Volatiles Lab:	☐

Non-Conformance : _____

Initials/Date: _____

Corrective Action: _____

Initials/Date: _____

Preventative Action: _____

Due Date: _____ **By Whom:** _____

Client Notification: FAX / TEL / SDQ / WRT **By Whom:** _____ **Date/Time:** _____

QA Review:

QA Officer: _____ **Area Supervisor:** _____

FIGURE52.XLS

Figure 52

5.0 REFERENCES

- Methods for Chemical Analysis of Water and Wastes; EPA-600/4-79-020 revised March, 1983.
- Standard Methods for the Evaluation of Water and Wastewater; Current Edition, APHA, AWWA, WPCF.
- Handbook for Analytical Quality Assurance in Water and Wastewater Laboratories; EPA 600/4-79-019, March 1979.
- Physical and Chemical Methods for the Evaluation of Solid Waste; EPA-SW846 Third Edition.
- Guidelines Establishing Text Procedures for the Analysis of Pollutants; 40 CFR; Parts 100 to 149, Current Edition.
- Water and Environmental Technology", Annual Book of ASTM Standards , Volume 11.01; Section 11; Current Edition.
- Good Automated Laboratory Practices; USEPA Office of Adiminstation and Resource Management, December 28, 1990.

6.0 TABLES

All Tables are currently incorporated into the text of the manual.

7.0 APPENDICES

Appendix A - Employee Training Record

Appendix B - Equipment List

Appendix C - Example Quality Control Reports

Appendix D - Bottle and Preservative Requirements

APPENDIX A
Training

Employee

Record

TriMatrix Laboratories, Inc.

LABORATORY ANALYST CERTIFICATION FORM

NAME OF ANALYST: _____

PROCEDURE NUMBER_____

PROCEDURE NAME_____

I. Certification Steps:

- Complete Items 1) through 5) on Laboratory Analyst Training Checklist.
- Analyze at least four performance evaluation samples by the same procedures used to analyze actual test samples.
- Calculate the average recovery (x) and the standard deviation (s) for each analyte of interest expressed as percent recovery of the performance evaluation sample.
- For each analyte, compare s and x with the corresponding acceptance criteria for precision and accuracy, respectively, given in the QC Acceptance Criteria at the end of the analytical method. If s and x for all analytes of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual test samples can begin.
- If any individual s exceeds the precision limit or any individual x falls outside the range for accuracy, then the system performance is unacceptable for that analyte and the following corrective action is required:
- Locate and correct the source of the problem and repeat the analysis for all analytes of interest which failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system; if this occurs, locate and correct the source of the problem. Repeat the test until the s and x for all analytes of interest meet the acceptance criteria.
- Attach a copy of all supporting analytical data to the Laboratory Analyst Certification Form.

Results of Performance Evaluation Sample Analysis:

Sample ID	Test Result (% Recovery)	Statistical Analysis of Data
_____	_____	Σ _____
_____	_____	n _____
_____	_____	x _____
_____	_____	s _____
_____	_____	x + s _____
_____	_____	

Analytical Method Acceptance:
Criteria:
Accuracy (x) _____
Precision (x + s) _____

II. Analysis of Blanks

- Analyze two MPB, results must be < current established MDL.

The certification sample results are acceptable and the analyst is authorized to perform this procedure for one year from the date below.
Signed(Area Manager) :_____ Date: _____

TriMatrix Laboratories, Inc.

LABORATORY ANALYST TRAINING CHECKLIST

NAME OF ANLAYST_____

METHOD NUMBER_____

METHOD TITLE _____

_____

PROCEDURE NUMBER _____

PROCEDURE TITLE _____

_____

_____ 1) The analyst has read the method and the standard operating procedure.

_____ 2) The instructor has reviewed the method and the procedure with the analyst.

_____ 3) The instructor has performed a manual demonstration of the procedure.

_____ 4) The analyst has successfully performed the procedure under direct supervision.

_____ 5) The analyst has successfully performed the procedure without direct supervision.

_____ 6) The analyst has successfully analyzed at least four performance evaluation samples, following the steps listed on the Laboratory Analyst Certification Form.

_____ 7) The Laboratory Analyst Certification Form has been compiled and all supporting analytical data attached.

INSTRUCTOR _____ DATE _____

Form Labancert; Revision New; Effective Date 11-30-94.

TriMatrix
Laboratories, Inc.

LABORATORY ANALYST TRAINING CHECKLIST

NAME OF ANLAYST_____

METHOD NUMBER_____

METHOD TITLE _____

_____

PROCEDURE NUMBER _____

PROCEDURE TITLE _____

_____

_____ The analyst has read the method and the standard operating procedure as required for annual recertification.

ANALYST _____ DATE _____

INSTRUCTOR _____ DATE _____

Form Labancert; Revision New; Effective Date 2-1-95.

TriMatrix Laboratories, Inc.

Analytical Services

Employee Training Record

Name: _____ **Employee No:** _____

Date of Hire: __/__/__

Position: _____ **Lab Area:** _____

Primary Responsibilities: _____

_____

_____

_____

_____

Position Change (1): _____ **Lab Area:** _____ **Start Date:** __/__/__ **End Date:** __/__/__

Primary Responsibilities: _____

_____

_____

_____

Position Change (2): _____ **Lab Area:** _____ **Start Date:** __/__/__ **End Date:** __/__/__

Primary Responsibilities: _____

_____

_____

_____

Training General

Signatures/Dates

Name:_____

	Trainee:	Trainer:
Laboratory Introduction/Walk-Through	_____ __/__/__	_____ __/__/__
Company Benefits Review-Personnel Dept.	_____ __/__/__	_____ __/__/__
Safety-Chemical Hygiene Plan Review	_____ __/__/__	_____ __/__/__
Safety Exam	_____ __/__/__	_____ __/__/__
Safety Walk/Safety Equipment Review	_____ __/__/__	_____ __/__/__
Computer Network/Email Training	_____ __/__/__	_____ __/__/__
Laboratory Information Management System	_____ __/__/__	_____ __/__/__

Document Control No: _____

Training-Quality Assurance

Signatures/Dates

Name: _____

	Trainee:	Trainer:
QA Officer Interview/QA Manual Review	_____ __/__/__	_____ __/__/__
General QA Objectives-QC Types	_____ __/__/__	_____ __/__/__
Bench sheets/Control Windows/Qualifications	_____ __/__/__	_____ __/__/__
Chemical Inventory Program	_____ __/__/__	_____ __/__/__
Stock Standard Record Procedures	_____ __/__/__	_____ __/__/__
Instrument Maintenance Logs	_____ __/__/__	_____ __/__/__
Instrument Run Logs	_____ __/__/__	_____ __/__/__
Analyst Notebook Procedures	_____ __/__/__	_____ __/__/__
Data Recording and Changes	_____ __/__/__	_____ __/__/__
Data Review and Documentation	_____ __/__/__	_____ __/__/__

Document Control : _____

Training-Laboratory Specific

Signatures/Dates

Name: _____

	Trainee:	Trainer:
Introduction to Apparatus/Equipment	____/__/__	____/__/__
Paperwork Flow-Lims Training	____/__/__	____/__/__
SOP Review-General Format	____/__/__	____/__/__
SOP Review-Method Specific	____/__/__	____/__/__
Methods: _____	____/__/__	____/__/__
_____	____/__/__	____/__/__
_____	____/__/__	____/__/__
_____	____/__/__	____/__/__
_____	____/__/__	____/__/__
_____	____/__/__	____/__/__
_____	____/__/__	____/__/__

Document Control No: _____

Training-Analyst Certifications

Signatures/Dates

Name: _____

		Trainee:	**Trainer:**
Methods:	_____	_____ __/__/__	_____ __/__/__
	_____	_____ __/__/__	_____ __/__/__
	_____	_____ __/__/__	_____ __/__/__
	_____	_____ __/__/__	_____ __/__/__
	_____	_____ __/__/__	_____ __/__/__
	_____	_____ __/__/__	_____ __/__/__
	_____	_____ __/__/__	_____ __/__/__
	_____	_____ __/__/__	_____ __/__/__
	_____	_____ __/__/__	_____ __/__/__
	_____	_____ __/__/__	_____ __/__/__

TriMatrix Laboratories, Inc.

Analytical Services

APPENDIX B
Equipment List - Inorganic

Instrument Description	Manufacturer	Model No.	Year Purchased	Detector or Type	Primary Use	TriMatrix I.D. No.
Auto-Analyzer (dual channel)	Bran & Lubbe	TRAACS 800	1987	VIS	353.2/350.1	160
Auto-Analyzer (four channel)	Lachat	Quick Chem	1990	VIS	300 series EPA	161
Conductivity Meter	YSI	32	1986	-----	120.1/9050	162
FTIR Spectrophotometer	Perkin-Elmer	1600	1990	IR	418.1	163
pH/mv Meter	Orion	710A	1993	-----	150.1	164
pH/mw/ISE Meter	Orion	EA920	1991	-----	150.1	165
Spectrophotometer (UV-VIS)	Shimadzu	1604	1990	UV/VIS	300 series EPA	166
Spectrophotomer (UV-VIS)	Shiamdzu	1201	1992	UV/VIS	300 series EPA	167
Total Organic Carbon Analyzer (TOC)	O.I.C.	700	1990	ND-IR	415.1	168
Total Organic Halogen Analyzer (TOX)	Xertex/Dohrman	---	1986	coulometric	9020	169
Turbidimeter	HACH	43900	1992	nephlometric	180.1	170
Polarograph	EG&G Princeton AR	384B	1991	dropping Hg	7198/8411	171

TriMatrix Laboratories, Inc.

Analytical Services

APPENDIX B
Equipment List - Inorganic

Auto-Titrator	Metler	DL12	1992	-----	310.x	-----

TriMatrix Laboratories, Inc.

Analytical Services

Equipment List - Atomic Absorption/ICP

Instrument Description	Manufacturer	Model No.	Year Purchased	Detector or Type	Primary Use	EARTH TEC I.D. No.
AA Spectrophotometer (flame/furnace)	Perkin Elmer	5100 PC	1989	190-800nm	200/7000 EPA	104
AA Spectrophotometer (furnace)	Perkin Elmer	5100 PC	1990	190-800nm	200/7000 EPA	105
AA Spectrophotometer (furnace)	Perkin Elmer	4100 PC	1992	190-800nm	200/7000 EPA	106
ICP Spectrophotometer	Perkin Elmer	Plasma 400	1989	160-800nm	200.7/6010	102
ICP Spectrophotometer	Perkin Elmer	Optima 3000	1993	167-792nm	200.7/6010	101
Autosampler	Perkin Elmer	AS-50	1989	-----	-----	-----
Autosampler	Perkin Elmer	AS-51	1990	-----	-----	-----
Autosampler	Perkin Elmer	AS-60	1992	-----	-----	-----
Autosampler	Perkin Elmer	FIAS-200	1989	-----	-----	-----
Mercury Amalgam System	Perkin Elmer	---	1990	-----	245.1	-----
Automated Mercury Analyzer	Leeman Labs	PS200	1992	-----	200/7000 EPA	103
Automated Mercury Preparation System	Leeman Labs	AP200	1992	-----	200/7000 EPA	-----
Microwave Digestion System	CEM	MDS 810	1989	-----	3051	-----

TriMatrix Laboratories, Inc.
Analytical Services

Equipment List - Gas Chromatography

Instrument Description	Manufacturer	Model No.	Year Purchased	Detector or Type	Primary Use	TriMatrix I.D. No.
Gas Chromatographs w/ECD	Varian	3700	1986	ECD	8080/8121	143
Gas Chromatograph w/FID	Varian	3400	1991	ECD/FID	8080	156
Gas Chromatographs w/ECD	Varian	3700	1988	FID/FID	8015/18-Air	130
Gas Chromatographs w/FID/ECD	Varian	3700	1988	FID/PID	8015/18-Air	131
Gas Chromatograph w/FID/CED	Varian	3400	1989	ECD/FID	8080	148
Gas Chromatograph w/Hall-PID	Tracor	540	1990	HALL/PID	601/602/8021	123
Gas Chromatograph w/Hall-PID	Varian	3400	1991	HALL/PID	601/602/8021	146/147
Gas Chromatograph w/Hall-PID	Tracor	9000	1992	HALL/PID	601/602/8021	125/126
Gas Chromatograph w/Hall-PID	Tracor	9000	1992	HALL/PID	601/602/8021	121/122
Autosampler	Varian	2016/LCS 2000	1988	-----	5030	-----
Autosampler	Varian	8000	1991	-----	5030	-----
Autosampler/Concentrator	Tekmar	2016/LCS 2000	1989	-----	5030	-----
Autosampler/Concentrator	Tekmar	2016/LCS 2000	1989	-----	5030	-----
Autosampler/Concentrator	Tekmar	2016/LCS 2000	1990	-----	5030	-----

TriMatrix Laboratories, Inc.
Analytical Services

Equipment List - Gas Chromatography

Autosampler	Tekmar	ALS 2050	1989	-----	5030	-----
Autosampler Concentrator	Tekmar	2016/LCS 2000	1989	-----	5030	-----
Autosampler/Concentrator	Tekmar	2016/LCS 2000	1989	-----	5030	-----
Autosampler/Concentrator	Tekmar	2016/LCS 2000	1991	-----	5030	-----
					5030	
Thermal Tube Desorber	Envirochem, Inc.	850	1990	-----	-----	-----

TriMatrix Laboratories, Inc.
Analytical Services

Equipment List - Gas Chromatography

Instrument Description	Manufacturer	Model No.	Year Purchased	Detector or Type	Primary Use	TriMatrix I.D. No.
Field GC's:						
Gas Chromatograph (FID/ECD)	Trimetrics	9000	1992	FID/ECD	Field Screen	128
Gas Chromatograph w/PID	HNU	301	1986	PID	Field Screen	-----
Gas Chromatograph (Hall/PID)	SRI	8610	1993	HALL/PID	Field Screen	-----
Gas Chromatograph (PID)	Photovac	10555	1991	PID	Field Screen	-----

TriMatrix Laboratories, Inc.

Analytical Services

Equipment List - HPLC

Instrument Description	Manufacturer	Model No.	Year Purchased	Detector or Type	Primary Use	TriMatrix I.D. No.
HPLC	Isco	2300	1988	UV	misc.	152
HPLC	Perkin Elmer	Series 410	1992	Diode Array	8310	151
LC Oven	Perkin Elmer	101	1992	-----	-----	-----
Diode Array Detector	Perkin Elmer	235	1992	-----	-----	-----
Fluorescence Detector	Perkin Elmer	LC240	1992	-----	-----	-----
Chromatography Data Systems:						
Turbochrom	Perkin Elmer		Continually Updated	-----	-----	-----

TriMatrix Laboratories, Inc.

Analytical Services

Equipment List - GC/MS

Instrument Description	Manufacturer	Model No.	Year Purchased	Detector or Type	Primary Use	TriMatrix I.D. No.
Ion Trap	Varian	Saturn II	1991	ION Trap	624/8260	132
Ion Trap	Varian	Saturn II	1991	ION Trap	624/8260	133
Ion Trap	Varian	Saturn II	1993	ION Trap	625/8270	138
Mass Spectrometer	Extrel	ELQ-400	1988	Quadrapole	625/8270	134
Mass Spectrometer	Extrel	ELQ-400	1989	Quadrapole	625/8270	135
Autosampler	Leap	CTC A2005	1993	-----	-----	-----
Autosampler	Leap	CTC A2005	1993	-----	-----	-----
Autosampler	Leap	CTC A2005	1993	-----	-----	-----
Gas Chromatographs w/FID(screening)	Varian	3700	1987	ECD/FID	8080	156
Autosampler/Concentrator	Tekmar	2016/LCS 2000	1991	-----	5030	-----
Autosampler	Dynatech	Dynasoils	1992	-----	5030	-----
Autosampler	Dynatech	Dynasoils	1992	-----	5030	-----
Autosampler	Dynatech	Dynasoils	1992	-----	5030	-----

Chromatography Data Systems:

TriMatrix Laboratories, Inc.

Analytical Services

Equipment List - GC/MS

Turbochrome	Perkin Elmer	4.0	1993	-----	-----	-----

APPENDIX C
Example
Quality Control Reports

15m7 J66t15m7 J66t

16-AUG-1994

ANALYTICAL BATCH DETAIL REPORT
INORGANIC LABORATORY

Batch Number: 71637 Date opened: 12-AUG-1994 Last Used: 16-AUG-1994 Owner: GEH Sequence: 1 Complete: N Control: Y

Method	Subset Application	Subset Ref. Citation	Samples	ICB	ICV	LCS	CCB	CCV
OPHOS/COLOR/WTR	WTR	USEPA-365.2						

	Sequen	QC Batch	Samp	SUR-Seq	Date	Anal	Parameter	Appli	Ref Cit	Det. Lim.	Init Conc	Final Conc	Spike	Typ	%	LCL	UCL	QC	E	D
v	1.000	000000.000.00			12-AUG	GEH	PHOSPHORUS, ORTHO	WTR	USEPA-365.2	0.01		< 0.01		ICB	0.00		10			
v	2.000	000000.000.00		-	12-AUG	GEH	PHOSPHORUS, ORTHO	WTR	USEPA-365.2	0.01		0.025	0.025	ICV	100.00	85	115			
v	3.000	071637.000.00	94789	-	12-AUG	GEH	PHOSPHORUS, ORTHO	WTR	USEPA-365.2	0.01		< 0.01								
v	4.000	071637.000.00	94790	-	12-AUG	GEH	PHOSPHORUS, ORTHO	WTR	USEPA-365.2	0.01		< 0.01								
v	5.000	071637.000.00	94791	-	12-AUG	GEH	PHOSPHORUS, ORTHO	WTR	USEPA-365.2	0.01		< 0.01								
v	6.000	000000.000.00		-	12-AUG	GEH	PHOSPHORUS, ORTHO	WTR	USEPA-365.2	0.01		< 0.01		CCB1	0.00		25			
v	7.000	000000.000.00		-	12-AUG	GEH	PHOSPHORUS, ORTHO	WTR	USEPA-365.2	0.01		0.49	0.5	CCV1	98.00	85	115			
v	8.000	000000.000.00		-	12-AUG	GEH	PHOSPHORUS, ORTHO	WTR	USEPA-365.2	0.01		< 0.01		BLK1	0.00					
v	9.000	000000.000.00		- 8	12-AUG	GEH	PHOSPHORUS, ORTHO	WTR	USEPA-365.2	0.01	< 0.01	0.40	0.38	LCS1	105.26	89.16	112.14			
v	10.000	071637.000.00	94791	- 5	12-AUG	GEH	PHOSPHORUS, ORTHO	WTR	USEPA-365.2	0.01	< 0.01	0.53	0.5	SPK1	106.00	88.02	117.42			
v	11.000	071637.000.00	94791	- 5	12-AUG	GEH	PHOSPHORUS, ORTHO	WTR	USEPA-365.2	0.01	< 0.01	< 0.01		DUP	0.00		20.88			

Batch Narrative

Analysis Narrative

94789	PHOSPHORUS, ORTHO	USEPA-365.2	WTR	16-AUG-94	DHM Sample was filtered prior to ortho phosphorus analysis.
94790	PHOSPHORUS, ORTHO	USEPA-365.2	WTR	16-AUG-94	DHM Sample was filtered prior to ortho phosphorus analysis.
94791	PHOSPHORUS, ORTHO	USEPA-365.2	WTR	16-AUG-94	DHM Sample was filtered prior to ortho phosphorus analysis.

QC BATCH QUALIFIERS

PARAM. NUM.	METHOD NUM.	METHOD VAR.	PARAMETER	METHOD	TYPE	RANGE			
1268	39	12	DECACHLOROBIPHENYL-SUR	GC/ECD/P&P/WW	SUR	1	45	88.470	34.940
1108	39	21	HEXABROMOBENZENE-PCB-SUR	GC/ECD/P&P/WTR	SUR	1	228	105.100	21.540
1267	39	21	TETRACHLORO-M-XYLENE-SUR	GC/ECD/P&P/WTR	SUR	1	139	97.750	18.760
1268	39	21	DECACHLOROBIPHENYL-SUR	GC/ECD/P&P/WTR	SUR	1	137	91.880	31.780
1108	39	23	HEXABROMOBENZENE-PCB-SUR	GC/ECD/P&P/SOIL	SUR	1	237	87.730	25.220
1268	39	23	DECACHLOROBIPHENYL-SUR	GC/ECD/P&P/SOIL	SUR	1	126	121.830	40.410
1268	39	24	DECACHLOROBIPHENYL-SUR	GC/ECD/P&P/SLG	SUR	1	3	160.330	24.090
1108	39	25	HEXABROMOBENZENE-PCB-SUR	GC/ECD/P&P/WST	SUR	1	45	99.330	13.590
1268	39	25	DECACHLOROBIPHENYL-SUR	GC/ECD/P&P/WST	SUR	1	9	163.220	37.980
1268	39	26	DECACHLOROBIPHENYL-SUR	GC/ECD/P&P/TCLP	SUR	1	19	139.890	23.560
1102	48	3	D5-NITROBENZENE-SUR	SEMI-VOL/MS/WW	SUR	1	135	71.750	12.790
1105	48	3	O-TERPHENYL-SUR	SEMI-VOL/MS/WW	SUR	1	135	81.030	14.690
1099	50	1	2-FLUOROPHENOL-SUR	SEMI-VOL/MS/WTR	SUR	1	781	43.940	9.280
1100	50	1	D6-PHENOL-SUR	SEMI-VOL/MS/WTR	SUR	1	777	29.240	6.860
1101	50	1	2,4,6-TRIBROMOPHENOL-SUR	SEMI-VOL/MS/WTR	SUR	1	772	74.720	14.210
1102	50	1	D5-NITROBENZENE-SUR	SEMI-VOL/MS/WTR	SUR	1	1212	70.740	11.090
1103	50	1	2-FLUOROBIPHENYL-SUR	SEMI-VOL/MS/WTR	SUR	1	1214	73.880	11.190
1105	50	1	O-TERPHENYL-SUR	SEMI-VOL/MS/WTR	SUR	1	1200	81.530	15.030
1099	50	2	2-FLUOROPHENOL-SUR	SEMI-VOL/MS/SOL	SUR	1	445	57.050	10.920
1100	50	2	D6-PHENOL-SUR	SEMI-VOL/MS/SOL	SUR	1	445	59.980	11.980
1101	50	2	2,4,6-TRIBROMOPHENOL-SUR	SEMI-VOL/MS/SOL	SUR	1	440	63.540	14.690
1102	50	2	D5-NITROBENZENE-SUR	SEMI-VOL/MS/SOL	SUR	1	1182	62.090	12.000
1103	50	2	2-FLUOROBIPHENYL-SUR	SEMI-VOL/MS/SOL	SUR	1	1227	66.510	11.880
1105	50	2	O-TERPHENYL-SUR	SEMI-VOL/MS/SOL	SUR	1	1206	72.820	14.080
1099	50	4	2-FLUOROPHENOL-SUR	SEMI'S/MS/TCLP	SUR	1	189	46.250	12.870
1100	50	4	D6-PHENOL-SUR	SEMI'S/MS/TCLP	SUR	1	185	31.200	9.800
1117	50	4	2,4,5-T-SUR	SEMI'S/MS/TCLP	SUR	1	48	64.420	16.090
1101	50	7	2,4,6-TRIBROMOPHENOL-SUR	SEMI'S/MS/SPLP	SUR	1	120	86.830	18.060
1102	50	7	D5-NITROBENZENE-SUR	SEMI'S/MS/SPLP	SUR	1	130	84.780	15.010
1103	50	7	2-FLUOROBIPHENYL-SUR	SEMI'S/MS/SPLP	SUR	1	130	85.160	15.160
1105	50	7	O-TERPHENYL-SUR	SEMI'S/MS/SPLP	SUR	1	130	84.630	15.760
1147	327	1	2,4-DB-SUR	GC/ECD/HRB/WTR	SUR	1	2	155.500	0.710

*** END OF REPORT ***

PARM NUM	METH NUM	METH VAR	PARAMETER	METHOD	REF CIT	TYPE	RANGE
1107	39	11	HEXABROMOBENZENE-PEST-SUR	GC/ECD/P&P/WTR	USEPA-608	SUR	1

BATCH	SEQ	F SEQ	SAMPLE	PENDING QTY	PENDING MEAN	PENDING STD DEV	PERCENT	CALCULATED T VALUE	STORED T VALUE
65053	2	003	82239	6	77.00	17.93	106.00	1.617	1.82
STATS AFTER EXCLUSION:				6	77.00	17.93			

PARM NUM	METH NUM	METH VAR	PARAMETER	METHOD	REF CIT	TYPE	RANGE
1107	39	12	HEXABROMOBENZENE-PEST-SUR	GC/ECD/P&P/WW	USEPA-608	SUR	1

BATCH	SEQ	F SEQ	SAMPLE	PENDING QTY	PENDING MEAN	PENDING STD DEV	PERCENT	CALCULATED T VALUE	STORED T VALUE
71878	2	002	93854	41	87.02	18.33	40.00	2.565	2.87
STATS AFTER EXCLUSION:				41	87.02	18.33			

PARM NUM	METH NUM	METH VAR	PARAMETER	METHOD	REF CIT	TYPE	RANGE
1108	39	12	HEXABROMOBENZENE-PCB-SUR	GC/ECD/P&P/WW	USEPA-608	SUR	1

BATCH	SEQ	F SEQ	SAMPLE	PENDING QTY	PENDING MEAN	PENDING STD DEV	PERCENT	CALCULATED T VALUE	STORED T VALUE
70863	63	001	93376	79	93.92	22.97	30.00	2.782	3.12
STATS AFTER EXCLUSION:				79	93.92	22.97			

PARM NUM	METH NUM	METH VAR	PARAMETER	METHOD	REF CIT	TYPE	RANGE
1267	39	12	TETRACHLORO-M-XYLENE-SUR	GC/ECD/P&P/WW	USEPA-608	SUR	1

BATCH	SEQ	F SEQ	SAMPLE	PENDING QTY	PENDING MEAN	PENDING STD DEV	PERCENT	CALCULATED T VALUE	STORED T VALUE
76272	20	002	101498	39	83.77	20.15	32.00	2.569	2.85
STATS AFTER EXCLUSION:				39	83.77	20.15			

PARM NUM	METH NUM	METH VAR	PARAMETER	METHOD	REF CIT	TYPE	RANGE
1268	39	12	DECACHLOROBIPHENYL-SUR	GC/ECD/P&P/WW	USEPA-608	SUR	1

BATCH	SEQ	F SEQ	SAMPLE	PENDING QTY	PENDING MEAN	PENDING STD DEV	PERCENT	CALCULATED T VALUE	STORED T VALUE
78943	10	001	106893	45	88.47	34.94	170.00	2.333	2.91

TRIMATRIX LABORATORIES, INC. - GRR

QC COMPLIANCE SUMMARY
Analytical Batches opened between 08-JAN-1996 and 22-JAN-1996
(result types BLK, CCB, CCV, CRL, ICB, ICV, IEC, LCS)

METALS LABORATORY
ANALYST: J.T. Whitmore

	Batches #	Batches %	Results #	Results %
Last used:	24		150	
Incomplete:	24	100.0	150	100.0
Out of warning:	0	0.0	0	0.0
Out of control:	0	0.0	0	0.0

*** THE FOLLOWING RESULTS ARE OUT OF CONTROL ***

RESULTS OUT OF CONTROL
THAT WERE RUN BETWEEN 19-Jan-1996 AND 19-Jan-1996

Lab Id: IN

Parameter	Method	Ref Cit	Appl	Anly	Batch Num	Seq	F Seq	Type		Sample	Percent
SULFATE	SO4/AUTO/WTR	USEPA-375.4	WATER	HLB	97724	24	0	SPK	2	136055	16

Parameter	Method	Ref Cit	Appl	Qualifier	Batch Num	Seq	F Seq	Qc Batch Num
SULFATE	SO4/AUTO/WTR	USEPA-375.4	WATER	2	97724	15	0	15709
SULFATE	SO4/AUTO/WTR	USEPA-375.4	WATER	33	97724	15	0	15709

TRIMATRIX LABORATORIES, INC. - GRR
PRE-DEFINED QUALIFIERS

Usage: N Description: NARRATIVE

Type: Q Description: QC BATCH

#	Explain	Active	Res Type	Repl	CLP	Site	Description
54	N	Y					The laboratory fortified blank (LFB) recovery for this analysis, fell outside the laboratory established control limits but within the test specification for this method. No data qualifications are required.

Usage: N Description: NARRATIVE

Type: R Description: RESULT

#	Explain	Active	Res Type	Repl	CLP	Site	Description
26	N	Y					All positive results for this sample and analysis were confirmed by a replicate measurement.
32	N	Y					The reporting limit for this analysis was elevated due to chromatographic interferences.
33	N	Y					This sample was filtered to remove particulate interferences prior to analysis.
37	N	Y					The procedure for the analysis of Cyanide Reactivity was not performed on this sample because the corresponding Total Cyanide result is <250 mg/kg.
38	N	Y					The procedure for the analysis of Amenable Cyanide was not performed on this sample because the corresponding Total Cyanide result is <0.005 mg/l.
39	N	Y					The procedure for the analysis of Amenable Cyanide was not performed on this sample because the corresponding Total Cyanide result is <0.05 mg/l.
40	N	Y					The procedure for the analysis of Free Cyanide was not performed on this sample because the corresponding Total Cyanide result is <0.005 mg/l.
41	N	Y					The procedure for the analysis of Free Cyanide was not performed on this sample because the corresponding Total Cyanide result is <0.05 mg/l.
42	N	Y					The reported Biochemical Oxygen Demand result for this sample was calculated on an oxygen depletion of <2.0 mg/l

PRE-DEFINED QUALIFIERS

Usage: N Description: NARRATIVE

Type: R Description: RESULT

#	Explain	Active	Res Type	Repl	CLP	Site	Description
43	N	Y					Nitrite Nitrogen was analyzed on this sample within the mandated USEPA 48 hour holding time. The sample was also chemically preserved and later analyzed for Nitrate+Nitrite Nitrogen within the 28 day USEPA mandated hold time. Nitrate Nitrogen was calculated by subtraction (NO3+NO2-NO2=NO3).
44	N	Y					The secondary surrogate (tetrachloro-m-xylene) % recovery for this sample fell outside the laboratory established control limits. The primary surrogate (decachlorobiphenyl) % recovery for this sample fell within the laboratory established control limits. No qualifications are required.
45	N	Y					The primary surrogate (decachlorobiphenyl) % recovery for this sample fell outside the laboratory established control limits. The secondary surrogate (terachloro-m-xylene) % recovery for this sample fell within the laboratory established control limits. No qualifications are required.
48	N	Y					This sample was filtered after distillation or digestion and prior to analysis, to remove a particulate interference.
55	N	Y					This sample was prepared in accordance to the Methanol sample extraction procedure, as outlined in USEPA Method 5030.
56	Y	Y					This sample also contains Cis-1,2-Dichloroethylene at a concentration of:
59	N	Y					This analysis was performed beyond an established USEPA maximum allowable parameter hold time. However, because the sample is a laboratory trip blank, the established hold time is inappropriate to the purpose of this analysis.

Usage: Q Description: QUALIFIER

Type: A Description: ANALYTICAL BATCH

#	Explain	Active	Res Type	Repl	CLP	Site	Description
4	N	Y			J	GRR	The laboratory control sample (LCS) recovery for this analysis, fell outside the laboratory established control limits. All samples in this analytical batch must be considered estimated.
49	N	Y					Analytical Batch Qualifier without Explanation.

TRIMATRIX LABORATORIES, INC. - GRR
PRE-DEFINED QUALIFIERS

Usage: Q Description: QUALIFIER

Type: Q Description: QC BATCH

#	Explain	Active	Res Type	Repl	CLP	Site	Description
50	Y	Y					See explanation below:
51	N	Y					QC Without

PRE-DEFINED QUALIFIERS

Usage: Q Description: QUALIFIER

Type: R Description: RESULT

#	Explain	Active	Res Type	Repl	CLP	Site	Description
1	Y	Y				GRR	Analysis performed beyond established USEPA maximum allowable parameter hold time. Result must be considered estimated.
2	N	Y	SPK	3	J	GRR	The matrix spike recovery (SPK) for this sample, fell outside the laboratory established control limits for this method and matrix. The corresponding sample result must be considered estimated.
3	N	Y	MSD			GRR	The matrix spike duplicate (MSD) RPD for this sample, fell outside the laboratory established control limits for this method and matrix. The corresponding sample result must be considered estimated.
5	N	Y				GRR	The laboratory fortified blank (LFB) recovery for this analysis, fell outside the laboratory established control limits. All samples in the associated pre-treatment batch must be considered estimated.
6	N	Y	DUP			GRR	The matrix duplicate (DUP) RPD for this sample, fell outside the laboratory established control limits for this method and matrix. The corresponding sample result must be considered estimated.
7	Y	Y				GRR	The reporting limit for this sample and the corresponding analysis is elevated due to an analytical interference which was a direct result of the sample matrix.
8	N	Y			U	GRR	Compound was not detected.
9	N	Y			J	GRR	Concentration is below the compound reporting limit, result must be considered estimated.
10	N	Y			M	GRR	Duplicate injection precision not met for this analysis.

PRE-DEFINED QUALIFIERS

Usage: Q Description: QUALIFIER

Type: R Description: RESULT

#	Explain	Active	Res Type	Repl	CLP	Site	Description
11	Y	Y				GRR	Sample integrity suspect upon arrival.
12	N	Y				GRR	Sample was received outside USEPA maximum allowable hold time. Result(s) must be considered estimated.
13	N	Y				GRR	Sample was lost during the sample preparation process, re-preparation of the sample was not possible due to low sample volume. The corresponding result(s) are not available.
14	N	Y				GRR	Sample was consumed during analysis, re-analysis was not possible due to low sample volume.
15	N	Y			J	GRR	Surrogate spike result(s) for this sample and analysis had a percent recovery outside the upper control limit for this method and matrix. All positive results must be considered estimated.
16	N	Y			J	GRR	Surrogate spike result(s) for this sample and analysis had a recovery of) 10%, but are below the lower control limit for this method and matrix. All positive results must be considered estimated. All (or non-dectectable results must be considered approximate.
17	N	Y			J	GRR	Surrogate spike result(s) for this sample and analysis had a recovery of (10%. All positive results must be considered estimated.
18	N	Y			UJ	GRR	Surrogate spike result(s) for this sample and analysis had a recovery of (10%. All (or non-detectable results must be considered unusable.
19	N	Y			J	GRR	Surrogate result(s) are unavailable due to a sample matrix interference p 'em encountered during the analysis of this sample. All p ve results must be considered estimated, all (or non- table results must be considered unusable.

PRE-DEFINED QUALIFIERS

Usage: Q Description: QUALIFIER

Type: R Description: RESULT

#	Explain	Active	Res Type	Repl	CLP	Site	Description
20	N	Y				GRR	Surrogate results are unavailable due to positive results in the sample extract resulting in a dilution of greater than 1:5 of the sample extract.
21	N	Y			U	GRR	The analysis of the Method Preparation Blank (MPB) for this parameter, method and associated digestion/extraction batch, had a positive value above the project reporting limit. This corresponding sample result is < 5 times the positive MPB value and therefore must be be considered estimated.
22	N	Y					The reporting limit for this sample and corresponding analysis is elevated due to the percent solids content of this sample.
23	N	Y					Matrix QC results (SPK and MSD) for this sample, QC batch, matrix and analysis were diluted out due to background matrix interferences.
24	N	Y					Matrix QC results (SPK and MSD) for this sample, QC batch, matrix and analysis are unavailable due to high analyte concentrations resulting in a dilution of this sample and/or it's extract.
25	N	Y					Matrix QC results (SPK and MSD) for this sample, QC batch, matrix and analysis are unavailable due to interfering peaks.
27	N	Y					The result(s) for this sample and analysis are reported on a "dry weight" basis.
28	N	Y					The result(s) for this sample and analysis are reported on an "as received" weight basis.
29	N	Y					As specified in 40CFR Part 136, Appendix A; Method 624, Acrolein and Acrylonitrile may only be screened by GC/MS. All positive results must therefore be considered estimated.

PRE-DEFINED QUALIFIERS

Usage: Q Description: QUALIFIER

Type: R Description: RESULT

#	Explain	Active	Res Type	Repl	CLP	Site	Description
30	N	Y					Sample was not preserved in accordance to 40 CFR Part 136.3; Table II; which states that a sample collected for Acrolein must be pH adjusted to a range of 4-5 or analyzed with 3 days of collection. All results for this compound must be considered estimated.
31	N	Y					The Analytical and/or Instrument Blank (BLK) for this parameter and method, had a positive value above the project reporting limit. This corresponding sample result is < 5 times the positive BLK value and therefore must be considered estimated.
34	N	Y					Total coliform bacterial colonies were detected in this sample. A confirmation procedure for the presence of E. coli was NEGATIVE.
35	N	Y					Total Coliform bacterial colonies were detected in this sample. A confirmation procedure for the presence of E. coli was POSITIVE.
36	N	Y					The reported detection limit for this sample and analysis is based on the current reagent water method detection limit (MDL).
46	N	Y			J		The percent difference (%D) between the original sample concentration and a serial dilution of this sample resulted in a value > 10%(D). Because the <10% difference criteria was not met, the corresponding sample and analysis must be considered estimated.
47	N	Y					The result for this analyte and method was reported at a value between the Method Detection Limit (MDL) and the laboratory established Reporting Limit. All postive results must be considered estimated, all non-detectable results must be considered aproximate.
52	N	Y					The result for this compound was quantitated from our continuing calibration standard but is estimated due to levels above the linear range of our calibration curve.
53	N	Y					Surrogate results are unavailable due to sample matrix interference(s); which resulted in a dilution of greater than 1:5 of the sample extract.

PRE-DEFINED QUALIFIERS

Usage: Q Description: QUALIFIER

Type: R Description: RESULT

#	Explain	Active	Res Type	Repl	CLP	Site	Description
57	N	Y					One or more surrogate compounds were not reported for this sample, matrix and method, due to unresolveable matrix intereferences. All remaining surrogate compound(s) had recoveries within the laboratory established control limits. No qualifications are required.
58	N	Y			N		The result for this sample and analysis was quantitated from the Photoionization Detector (PID) due to an interfering peak on the Electrolytic Conductivity Detector (HECD).
60	N	Y			J		The result for this compound was quantitated from our average response factor of the calibration curve but is estimated due to levels above the linear range of the curve.
61	N	Y			J		The reported result for this sample is the minimum estimated amount based on the upper limit of the calibration curve and all sample extraction and/or dilution factors.
62	N	Y	PDS	1	J		The post digestion spike result (PSD) for this sample had a percent recovery of outside the 85 - 115 % method window. All corresponding positive sample results where the PDS is <40% are estimated, all non-detectable results with a PDS <40 & 10% are considered approximate, all <10% are unusable.
63	N	Y	SPK				One of the matrix spike accuracies fell outside of the esatblished laboratory control limits, but the precision for MS/MSD is within the control limits for this matrix and method. The corresponding sample result is not considered estimated.
64	N	Y					The detection limit for this sample and corresponding analysis were elevated due to insufficient sample volume received.
65	N	Y					Matrix quality control analysis unavailable due to insufficient sample volume received.

APPENDIX D

SAMPLE RECEIVING AND STORAGE
BOTTLE AND PRESERVATION REQUIREMENTS

The collection of the sample is the starting point for the generation of quality data. It is the responsibility of TriMatrix to provide the client who collects the sample with sample collection instructions which ensure sample integrity. Also, where applicable TriMatrix also supplies the client with appropriate clean sample containers and preservative chemicals; these glass containers are purchased new and certified as clean and vendors such as I-Chem Research and Fischer Scientific.

Sampling and Preservation Requirements for certain common environmental analyses are listed in the following table: (NOTE: Holding times are based on EPA guidelines for CLP, NPDES, and RCRA).

APPENDIX D
SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Analyte	Matrix	Holding Time (fron Date Sampled)	Preservation	Container	Sample Size	Method Reference	Container Label
Volatile Halocarbons-GC **	Water	14 days	4°C	2-40ml VOA vials	40 ml	601 or 8010 or 8240 or 8260	Yellow
	Soil/Waste	14 days	4°C	Glass 4 oz. jar	40g		
Volatile Aromatics-GC **	Water	7 days 14 days	4°C 4°C w/Hcl	2-40 ml VOA vials	40 ml	602 or 8020 or 8240 or 8260	Yellow
	Soil/Waste	14 days	4°C	Glass 4 oz. jar or VOA vial	40 g		
Phenols **	Water	* 7 days	4°C	One liter glass bottle	1000 ml	604 or 8040 or 8270	Orange
	Soil/Waste	* 14 days	4°C	Glass 4 oz. jar	100 g		
Phthalate Esters	Water	* 7 days	4°C	One liter glass bottle	1000 ml	606 or 8060 or 8270	Orange
	Soil/Waste	* 14 days	4°C	Glass 4oz. jar	100 g		
Organo-chlorine Pesticides/ PCB's	Water	* 7 days	4°C	One liter glass bottle	1000 ml 8080	608 or	Orange
	Soil/Waste	* 14 days	4°C	Glass 4oz. jar	100 g		
	PCB oils	N/A	None	VOA Vial	20 ml		
Polyaromatic Hydrocarbons **	Water	* 7 days	4°C	One liter glass bottle	1000 ml	610 or 8100 or 8270 or 8310	Orange
	Soil/Waste	* 14 days	4°C	Glass 4 oz. jar	100 g		
Organo-phosphorous Pesticides	Water	* 7 days	4°C	One liter glass bottle	1000 ml	614 or 8140	Orange
	Soil/Waste	* 14 days	4°C	Glass 4 oz. jar	100 g		
Phenoxy Acid Herbicides	Water	* 7 days	4°C	One liter glass bottle	1000 ml	8150	Orange

APPENDIX D
SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

	Soil/Waste	* 14 days	4°C	Glass 4 oz. jar	100 g		
Volatiles-GC/MS **	Water	7 days 14 days	4°C 4°C w/Hcl	2-40 ml VOA vials	40 ml	8240 or 8260	Yellow
	Soil/Waste	14 days	4°C	Glass 4 oz. jar	100 g		

APPENDIX D
SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

1,2-Dibromoethane 1,2-Dibromo-3-chloropropane	Drinking Water	28 days	4°C	2-40ml VOA vials	40 ml	504	Orange
Semi-Volatiles	Water	* 7 days	4°C	One liter glass bottle	1000 ml	8270	Orange
**	Soil/Waste	* 14 days	4°C	Glass 4oz. jar	100 g		
Carbamate & Urea Pesticides	Water	* 7 days	4°C	One liter glass bottle	1000 ml	531.1	Orange
	Soil/Waste	* 14 days	4°C	Glass 4 oz. jar	100 g		
Dioxins/ Furans	Water	*7 days	4°C	One liter glass bottle	1000 ml	Screen-625	Orange
	Soil/Waste	None Required	4°C	Glass 4 oz. jar	100 g		
Petroleum Hydrocarbons	Water	*7 days	4°C H2SO4	One liter glass bottle	1000 ml	418.1 or 9071 or 9073 04 Cal. Method-D.R.	Green
	Soil/Waste	* 14 days	4°C	Glass 4 oz. jar	100 g		
BETX/TPH-GRO	Water	7 days	4°C	2-40ml VOA vials	40 ml	8020 or Modified 8015	Green
	Soil/Waste	14 days	4°C w/Hcl 4°C	Glass 4 oz. jar	100 g		
TCLP-UST (Benzene, TPH, Lead)	Soil/Waste	14 days	4°C	Glass 4oz. jar	100 g	1311	Green
Metals (ICP)	Water	6 months	HNO3 to pH <2.0	16 oz. plastic bottle	500 ml	260.7 or 6010	Red
	Soil/Waste	6 months	None	4 oz. glass jar	50 g		
Arsenic (GF-AA)	Water	6 months	HNO3 to pH <2.0	16 oz. plastic bottle	500 ml	7060 or 206.2	Red
	Soil/Waste	6 months	None	4 oz. glass jar	50 g		
Mercury (CV-AA)	Water	28 days	HNO3 to pH <2.0	16 oz. plastic bottle	500 ml	245.1 or 7470 or	Red
	Soil/Waste	28 days	None	4 oz. glass jar	50 g		

APPENDIX D
SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Selenium (GF-AA)	Water	6 months	HNO3 to pH <2.0	16 oz. plastic bottle	500 ml	7470 or 270.2	Red
	Soil/Waste	6 months	None	4 oz. glass jar	50 g		
Thallium (GF-AA)	Water	6 months	HNO3 to pH <2.0	16 oz. plastic bottle	500 ml	7421 or 279.2	Red
	Soil/Waste	6 months	None	4 oz. glass jar	50 g		
Lead (GF-AA)	Water	6 months	HNO3 to pH <2.0	16 oz. plastic bottle	500 ml	S.M. 3500 239.2	Blue
	Soil/Waste	6 months	None	4 oz. glass jar	50 g		
Chromium (III/VI)	Water	24 hours	4°C	16 oz. plastic bottle	500 ml	S.M. 3500 Cr D.	Blue
	Soil/Waste	24 hours	4°C	4 oz. glass jar	50 g		
Chromium (Total)	Water	6 months	HNO3 to pH < 2.0	16 oz. plastic bottle	500 ml	6010	Red
	Soil/Waste	6 months	None	4 oz. glass jar	50 g		
Silica	Water	28 days	4°C	16 oz. plastic bottle	500 ml	6010	Red
	Soil/Waste	28 days	4°C	4 oz. glass jar	50 g		
Color	Water	48 hours	4°C	8 oz. plastic bottle	250 m	S.M. 2120 B	Blue
Oil & Grease	Water	28 days	4°C H2SO4 to	1 liter glass bottle	1000 ul	S.M. 5520 B or 413.1 or	Blue
	Soil/Waste	14 days	None	4 oz. glass jar		9050 or 9071	
Specific Conductance	Water	28 days	4°C	8 oz. plastic bottle	250 ml	S.M. 2510 or 120.1 or 9050	Blue
Acidity	Water	14 days	4°C	8 oz. plastic bottle	250 ml	S.M. 2310 or 350.1	Blue
pH	Water	24 hours	4°C	8 oz. plastic	50 ml	S.M. 2400 H+ or	Blue

APPENDIX D
SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

	Soil/Waste	24 hours	4°C	bottle 4 oz. glass jar	100 g	150.1 or 9045/9041	
Alkalinity	Water	14 days	4°C	8 oz. plastic bottle	250 ml	S.M. 2320 or 310.1	Blue
Hardness	Water	6 months	HNO3 to pH <2.0	8 oz. plastic bottle	250 ml	S.M. 2340 B	Blue
Biochemical Oxygen Demand (BOD)	Water	48 hours	4°C	16 oz. plastic bottle bottle	500 ml	S.M. 521 B or 405.1	Blue
Chemical Oxygen Demand (COD)	Water	28 days	4°C H2SO4 to pH <2.0	8 oz. plastic bottle	250 ml	S.M. 5220 or 410.4	Blue
Organic Carbon (TOC)	Water	28 days	4°C H2SO4 to pH <2.0	8 oz. plas/bottle or 4 oz. glass jar or 2-VOA vials	250 ml 125 ml 80 ml	S.M. 5310 B or 415.1 or 9060	White
Chlorophyll	Water	7 days	4°C keep dark; foil wrap	500 ml plastic bottle	500 ml	S.M. 102OOH	Blue
Ortho-Phosphate	Water	48 hours	4°C	8 oz. plastic bottle	100 ml	S.M. 4500-PC or 300.0	Blue
Total Phosphorus	Water	28 days	H2SO4 to pH <2.0	8 oz. plastic bottle	250 ml	S.M. 4500-PC	Blue
Total Kjeldahl Nitrogen (TKN)	Water Soil/Waste	28 days 28 days	4°C H2SO4 to pH <2.0 None	8 oz. plastic bottle 4 oz. glass jar	250 ml 100 g	S.M. 4500-N (org) B or 351.4	Blue
Ammonia	Water	28 days	4°C H2SO4 to pH <2.0	8 oz. plastic	250 ml	S.M. 4500-NH3. F or 350.3	Blue

APPENDIX D
SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Nitrite	Water	48 hours	4°C	8 oz. plastic bottle	250 ml	S.M. 2400-NO2.C.	Blue
Nitrate	Water	48 hours	4°C	8 oz. plastic bottle	250 ml	S.M. 4500-NO3.C. or 300.0	Blue
Nitrite plus Nitrate (No distinction between NO2 and NO3)	Water	48 hours	4°C	8 oz. plastic bottle	250 ml	S.M. 4500-NO2, NO3.C or 300.0	Blue
Total Volatile	Water	7 days	4°C	8 oz. plastic bottle	250 ml	S.M. 2540-E or 160.4	Blue
Solids	Soil/Water	7 days	4°C	4 oz. glass jar	100 g		
Turbidity	Water	48 hours	4°C	8 oz. plastic bottle	250 ml	S.M. 2130	Blue
Sulfate	Water	28 days	4°C	8 oz. plastic bottle	250 ml	S.M. 4500-SO4 B.	Blue
Sulfite	Water	48 hours 3 ml 1% EDTA	4°C	8 oz. plastic	250 ml	S.M. 4500-SO3 B.	Blue
Sulfide	Water	7 days	4°C, NaOH, Zinc Acetate	8 oz. plastic bottle	250 ml	S.M. 4500-S2 E.	Blue
Acrolein **	Water	3 days	4°C	VOA vial	40 ml	624, 603, 8240 8260	Yellow
	Water	14 days	4°C, pH4-5	VOA vial	40 ml	624, 503, 8240, 9260	
Acrylonitrile **	Water	14 days	4°C	VOA vial	40 ml	524, 503, 8240, 8260	Yellow
	Water	14 days	4°C, pH4-5	VOA vial	40 ml	624, 603, 8240, 8260	
Cyanide **	Water	14 days	4°C, NaOH to pH >12	32 oz. plastic	1000 ml	S.M. 4500-CN E or 335.2 or	Blue

APPENDIX D
SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

	Soil/Waste	14 days	4°C	4 oz. glass jar	100 g	9010	
Coliform Fecal and Total **	Water	24 hours	4°C	Sterile plastic bottle or whirl pak	100 ml	S.M. 9222-B, D. or 9132	White
Bromide	Water	28 days	4°C	8 oz. plastic bottle	250 ml	S.M. 4500 Br-C. or 300.0	Blue
Chloride	Water	28 days	4°C	8 oz. plastic bottle	250 ml	S.M. 4500-Cl-B or S.M. 4500-Cl-F or 300.0	Blue
Chlorine Residual	Water	Analyze Immediately	4°C	8 oz. plastic bottle	250 ml	S.M. 4500 B.	Blue
Total Solids (% Moisture)	Water	7 days	4°C	8 oz. plastic bottle	250 ml 160.3	S.M. 2540 B. or	Blue
	Soil/Waste	7 days	4°C	4 oz. glass jar	100 g		
Total Dissolved Solids (TDS)	Water	7 days	4°C	8 oz. plastic bottle	250 ml	S.M. 2540 C. or 160.2	Blue
Total Suspended Solids (TSS)	Water	7 days	4°C	8 oz. plastic bottle	250 ml	S.M. 2540 D. or 160.1	Blue
TDS plus TSS	Water	7 days	4°C	16 oz. plastic bottle	250 ml	Same as Above	Blue
Fluoride	Water	28 days	None	8 oz. plastic bottle	250 ml	S.M. 2500F-C. or 340.2	Blue
Iodide	Water	28 days	None	8 oz. plastic bottle	250 ml	Ion Chromatograph Not EPA Approved	Blue
Organic Halogen	Water	7 days	4°C H2SO4 to	500 ml glass Bottle	560 ml	S.M. 5320 B 9020	White

APPENDIX D
SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

(TOX)			pH <2.0				
Phenolics	Water	28 days	4°C H2SO4 pH <2.0	1 liter glass bottle	1060 ml	S.M. 5530 C. or 420.1 or 9065	Blue
Surfactants (MBAS)	Water	48 hours	4°C	8 oz. plastic bottle	250 ml	S.M. 5540 C. or 425.1	Blue
TCLP-	Soil/Waste					1311	White
Volatiles		14 days	4oC	4 oz. glass jar	100 g		
Semi-Volatiles		14 days	4oC	8 oz. glass jar	250 g		
Metals		180 days (Hg-28 days)	None	8 oz. glass jar	250 g		
Pest./Herb.		14 days	4oC	8 oz. glass jar	250 g		
Flash Point	Solid/Liq./	N/A	None	(Appropriate to Sample)	250 g or	1010	White
	Waste	N/A	None	8 oz. glass jar or 8 oz. plastic (min. 0 ml) bottle	250 ml		
Corrosivity (pH and Method 1110)	Waste	N/A	None	(Appropriate to Sample) 16 oz. glass jar or 16 oz. plastic bottle	500 ml	9040, 9041	White
Paint Filter (Free Liquids)	Waste	N/A	None	(Appropriate to Sample) 4 oz. glass jar or 8 oz. plastic bottle	250 ml or 50 g	9095	White
Chloride/ Halogens (on Waste)	Waste	N/A	None	(Appropriate (to Sample) 4 oz. glass jar or 8 oz. plastic bottle	50 ml 50 g	ASTM D808	White
Radionuclides (Alpha + Beta, Alpha, Beta, Ra226, Ra228	Water	6 month	HNO3 to pH <2.0	32 oz. plastic bottle or 32 oz. glass bottle	1000 ml		White

APPENDIX D
SAMPLE COLLECTION GUIDELINES
BOTTLE AND PRESERVATIVE REQUIREMENTS

Reactivity (Releasable CN and S)	*Waste*	*N/A*	*4°C*	*(Appropriate to* Sample 8 oz. plastic bottle or 4oz. glass jar	*10 g*	*SW 846* Chapt. 7	*White*

* 7 or 14 days until extraction, 40 days after extraction

** Use Sodium Thiosulfate or Ascorbic Acid if samples are chlorinated

NOTE: For Organics parameters, container lid should be teflon or metal foil lined.

NOTE: For Inorganic parameters, container lid should be plastic or teflon lined.

NOTE: When testing for several like parameters (ICP metals, Ion Chromotograph anions), one container per sample is sufficient. For example, a sample to be tested for the 13 priority pollutant metals needs one 500 ml container.

8.0 GLOSSARY OF TERMS

ABSORBANCE - a measure of the decrease in incidient light passing through a sample into the detector. It is defined mathematically as:

$$A = \frac{I\,(solvent)}{I\,(solution)} - \log \frac{Io}{I}$$

ALIQUOT - a measured portion of a field sample taken for analysis.

ANALYSIS DATE/TIME - the date and military time (24-hour clock) of the introduction of the sample, standard, or blank into the analysis system.

ANALYTE - the element or ion an analysis seeks to determine; the element of interest.

ANALYTICAL SAMPLE - any solution or media introduced into an instrument on which an analysis is performed excluding instrument calibration, initial calibration verification, initial calibration blank, continuing calibration verification and continuing calibration blank. Note the following are all defined as analytical samples: undiluted and diluted samples (EPA and non-EPA), predigestion spike samples, duplicate samples, serial dilution samples, analytical spike samples, post-digestion spike samples, interference check samples (ICS), CRDL standard for AA (CRA), CRDL standard for ICP (CRI), laboratory control sample (LCS), method preparation blank (MPB) and linear range analysis sample (LRS).

ANALYTICAL SPIKE - The furnace post-digestion spike. The addition of know amount of standard after digestion.

AUTOZERO - zeroing the instrument at the proper wavelength. It is equivalent to running a standard blank with the absorbance set at zero.

AVERAGE INTENSITY - the average of two different injections (exposures).

BACKGROUND CORRECTION - a technique to compensate for variable background contribution to the instrument signal in the determination of trace elements.

BLANK - an analytical sample designed to assess specific sources of laboratory contamination. See individual types of Blanks: Method Blank, Instrument Blank, Storage Blank, and Sulfur Blank.

BAR GRAPH SPECTRUM - a plot of the mass-to-charge ratio (m/e) versus relative intensity of the ion current.

BATCH - a group of samples prepared at the same time in the same location using the same method.

BREAKDOWN - a measure of the decomposition of certain analytes (DDT and Endrin) into by-products.

4-BROMOFLUOROBENZENE (BFB) - the compound chosen to establish mass spectral instrument performance for volatile (VOA) analyses. It is also in the VOA fraction as a system monitoring compound (SMC).

CALIBRATION - the establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type of acid or concentration of acids as used in the sample preparation.

CALIBRATION BLANK - a volume of acidified deionized/distilled water.

CALIBRATION STANDARDS - a series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

CALIBRATION FACTOR (CF) - a measure of the gas chromatographic response of a target analyte to the mass injected. The calibration factor is analogous to the Relative Response Factor (RRF) used in the Volatile and Semivolatile fractions.

CASE - a finite, usually predetermined number of samples collected over a given time period from a particular site. Case numbers are assigned by the Sample Management Office. A Case consists of one or more Sample Delivery Groups.

CHARACTERIZATION - a determination of the approximate concentration range of compounds of interest used to choose the appropriate analytical protocols.

COEFFICIENT OF VARIATION (CV) - the standard deviation as a percent of the arithmetic mean.

CONCENTRATION LEVEL (low or medium) - characterization of soil samples or sample fractions as low concentration or medium concentration is made on the basis of the laboratory's preliminary screen, not on the basis of information entered by the sampler.

CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

CONTINUING CALIBRATION - analytical standard run ever 12 or 24 hours to verify the initial calibration of the system.

CONTINUOUS LIQUID-LIQUID EXTRACTION - used herein synonymously with the terms continuous extraction, continuous liquid extraction, and liquid extraction. This extraction technique involves boiling the extraction solvent in a flask and condensing the solvent above the aqueous sample. The condensed solvent drips through the sample, extracting the compounds of interest from the aqueous phase.

CONTRACT REQUIRED DETECTION LIMIT (CRDL) - minimum level of detection acceptable under the contract Statement of Work.

CONTROL LIMITS - a range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.,

CORRELATION COEFFICIENT - a number (r) which indicates the degree of dependence between two variables (concentration - absorbance). The more dependent they are the closer the value to one. Determined on the basis of the least squares line.

DATE - MM/DD/YY - where MM = 01 for January, 02 for February, ... 12 for December; DD = 01 to 31; YY = 94, 95, 96, 97, etc.

DAY - unless otherwise specified, day shall mean calendar day.

DIGESTION LOG - an official record of the sample preparation (digestion).

DISSOLVED METALS - analyte elements which have not been digested prior to analysis and which will pass through a 0.45 um filter.

DRY WEIGHT - the weight of a sample based on percent solids. The weight after drying in an oven.

DUPLICATE - a second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

EXTRACTED ION CURRENT PROFILE (EICP) - a plot of ion abundance versus time (or scan number) for ion(s) of specified mass(es).

EXTRACTABLE - a compound that can be partitioned into an organic solvent from the sample matrix and is amenable to gas chromatography. Extractables include semivolatile (BNA) and pesticide/Aroclor compounds.

FIELD BLANK - any sample submitted from the field identified as a blank.

FIELD SAMPLE - a portion of material received to be analyzed that is contained in single or multiple containers and identified by a unique Sample Number.

FLAME ATOMIC ABSORPTION (AA) - atomic absorption which utilizes flame for excitation.

GRAPHITE FURNACE ATOMIC ABSORPTION (GFAA) - atomic absorption which utilizes a graphite cell for excitation.

GAS CHROMATOGRAPH (GC) - the instrument used to separate analytes on a stationary phase within a chromatographic column. The analytes are volatized directly from the sample (VOA water and low-soil), volatized from the sample extract (VOA medium soil), or injected as extracts (SVOA and PEST). In VOA and SVOA analysis, the compounds are detected by a Mass Spectrometer (MS). In PEST analysis, the compounds are detected by an Electron Capture (EC) detector. In the screening procedure (all fractions), the Flame Ionization Detector (FID) is used as the detector.

GEL PERMEATION CHROMATOGRAPHY (GPC) - a size-exclusion chromatographic technique that is used as a cleanup procedure for removing large organic molecules, particularly naturally occurring macro-molecules such as lipids, polymers, viruses, etc.

HOLDING TIME - the elapsed time expressed in days from the date sampled by the Contractor until the date of its analysis.

Holding time - (sample analysis date - sample collection date)

INDEPENDENT STANDARD - a Contractor-prepared standard solution that is composed of analytes from a different source than those used in the standards for the initial calibration.

INDUCTIVELY COUPLED PLASMA (ICP) - a technique for the simultaneous or sequential multi-element determination of elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Characteristic atomic line emission spectra are produced by excitation of the sample in a radio frequency inductively coupled plasma.

IN-HOUSE - at the Contractor's facility.

INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the mass spectrometer or electron capture detector to the target compounds.

INJECTION - introduction of the analytical sample into the instrument excitation system for the purpose of measuring absorbance, emission or concentration of an analyte. May also be referred to as exposure.

INSTRUMENT CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

INSTRUMENT DETECTION LIMIT (IDL) - determined by multiplying by three the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration of 3x-5x IDL on three nonconsecutive days with seven consecutive measurements per day.

INSTRUMENT CHECK SAMPLE - a solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors.

INSTRUMENT CHECK STANDARD - a multi-element standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis.

INTEGRATION SCAN RANGE - this scan number of the scan at the beginning of the area of integration to the scan number at the end of the area of integration.

INTERFERENTS - substances which affect the analysis for the element of interest.

INTERNAL STANDARDS - compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for volatiles), and sample extract (for semivolatiles) at a known concentration, prior to analysis. Internal standards are used as the basis for quantitation of the target compounds.

INSTRUMENT/ANALYTICAL BLANK - a blank designed to determine the level of contamination associated with the analytical instruments.

INSUFFICIENT QUANTITY - when there is not enough volume (water sample) or weight (soil/sediment) to perform any of the required operations: sample analysis or extraction, percent moisture, MS/MSD, etc.

LABORATORY - synonymous with Contractor as used herein.

LABORATORY CONTROL SAMPLE (LCS) - a control sample of known composition. Aqueous and solid laboratory control samples are analyzed using the same sample preparation, reagents, and analytical methods employed for the samples received.

LABORATORY RECEIPT DATE - the date on which a sample is received at the Contractor's facility, as recorded on the shippers delivery receipt.

LINEAR RANGE, LINEAR DYNAMIC RANGE - the concentration range over which the ICP analytical curve remains linear.

MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this statement of work, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

MATRIX EFFECT - in general, the effect of a particular matrix (water or soil/sediment) on the constituents with which is contacts. This is particularly pronounced for clay particles which may adsorb chemicals and catalyze reactions. Matrix effects may prevent extraction of target analytes, and may affect surrogate recoveries. In addition, nontarget analytes may be extracted from the matrix causing interferences.

MATRIX MODIFIER - salts used in AA to lessen the effects of chemical interferents, viscosity, and surface tension.

MATRIX SPIKE - aliquot of a matrix (water or soil) fortified (spiked) with know quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - an analytical control consisting of all reagents, internal standards and surrogate standards (or SMCs for VOA), that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background and reagent contamination.

METHOD OF STANDARD ADDITIONS (MSA) - the addition of 3 increments of a standard solution (spikes) to sample aliquots of the same size. Measurements are made on the original and after each addition. The slope, x-intercept and y-intercept are determined by least-square analysis. The analyte concentration is determined by the absolute value of the x-intercept. Ideally, the spike volume is low relative to the sample volume (approximately 10% of the volume). Standard addition may counteract matrix effects; it will not counteract special effects. Also referred to as Standard Addition.

m/z - Mass to charge ration, synonymous with "m/e"

NARRATIVE - portion of the data package which includes laboratory, contract, case and sample number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.

PERCENT DIFFERENCE (%D) - to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference).

PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105°C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105°C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

PERCENT SOLIDS - the proportion of solid in a soil sample determined by drying an aliquot of the sample.

PERFORMANCE EVALUATION MIXTURE - a calibration solution of specific analytes used to evaluate both recovery and percent breakdown as measures of performance.

PERFORMANCE EVALUATION (PE) SAMPLE - a sample of known composition provided by EPA for Contractor analysis. Used by EPA or client to evaluate laboratory performance.

PREPARATION BLANK (reagent blank, method blank) - an analytical control that contains distilled, deionized water and reagents, which is carried through the entire analytical procedure (digested/distilled or extracted and analyzed). An aqueous method blank is treated with the same reagents as a sample with a water matrix; A solid method blank is treated with the same reagents as a soil sample.

PRIMARY QUANTITATION ION - a contract specified ion used to quantitate a target analyte.

PROTOCOL - a compilation of the procedures to be followed with respect to sample receipt and handling, analytical methods, data reporting and deliverables, and document control. Used synonymously with Statement of Work (SOW).

PURGE AND TRAP (DEVICE) - analytical technique (device) used to isolate volatile (purgeable) organics by stripping the compounds from water or soil by a stream of inert gas, trapping the compounds on an adsorbent such as a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column.

PURGEABLES - volatile compounds.

QUALITY CONTROL SAMPLE - a solution obtained from an outside source having known concentration values to be used to verify the calibration standards.

REAGENT BLANK - a volume of deionized, distilled water containing the same acid matrix as the calibration standards carried through the entire analytical scheme.

REAGENT WATER - water in which an interferent is not observed at or above the minimum quantitation limit of the parameters of interest.

RECONSTRUCTED ION CHROMATOGRAM (RIC) - a mass spectral graphical representation of the separation achieved by a gas chromatograph; a plot of total ion current versus retention time.

RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOW and elsewhere to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. (In contrast, see percent difference above).

RELATIVE RESPONSE FACTOR (RRF) - a measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RRF is determined by the following equation:

$$RRF = \frac{Ax}{Ais} \times \frac{Cis}{Cx}$$

where:
A = area of the characteristic ion measured
C = concentration
is = internal standard
x = analyte of interest

RELATIVE RETENTION TIME (RRT) - the ration of the retention time of a compound to that of a standard (such as an internal standard).

$$RRT = \frac{RTc}{RTis}$$

Where,

RTc = Retention time for the semivolatile target or surrogate compound in continuing calibration.
RTis = Retention time for the internal standard in calibration standard or in a sample.

RESOLUTION - also termed separation or percent resolution, the separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

RESOLUTION CHECK MIXTURE - a solution of specific analytes used to determine resolution of adjacent peaks; used to assess instrumental performance.

RESPONSE - or Instrumental Response: a measurement of the output of the GC detector (MS, EC, or FID) in which the intensity of the signal is proportionate to the amount (or concentration) detected. Measured by peak area or peak height.

RETENTION TIME (RT) - the time a target analyte is retained on a GC column before elution. The identification of a target analyte is dependent on a target compound's retention time falling within the specified retention time window established for that compound. Retention time is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

ROUNDING RULES - If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. As an example, 11.443 is rounded off to 11.44.

If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1. As an example, 11.446 is rounded off to 11.45.

If the figure following those to be retained is 5, and if there are no figures other than zeros beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number. As an example, 11.435 is rounded off to 11.44, while 11.425 is rounded off to 11.42.

If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures.

See forms instructions (Exhibit B) for exceptions.

RUN - a continuous analytical sequence consisting of prepared samples and all associated quality assurance measurements as required by the contract Statement of Work.

SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

* Case; or
* Each 20 samples within a Case; or
* Each 14-day calendar period during which samples in a Case are received, beginning with receipt of the first sample in the Case or SDG.

Samples may be assigned to Sample Delivery Groups by matrix (i.e., all soils in one SDG, all waters in another), at the discretion of the laboratory.

SAMPLE NUMBER - a unique identification number designated for each sample. The Sample Number appears on all laboratory documents which contain information on that sample.

SEMIVOLATILE COMPOUNDS - compounds amenable to analysis by extraction of the sample with an organic solvent. Used synonymously with Base/Neutral/Acid (BNA) compounds.

SENSITIVITY - the slope of the analytical curve, i.e., functional relationship between emission intensity and concentration.

SERIAL DILUTION - the dilution of a sample by a factor of five. When corrected by the dilution factor, the diluted sample must agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

SOIL - synonymous with soil/sediment or sediment as used herein.

SONIC CELL DISRUPTOR (SONICATOR) - a device that uses the energy from controlled ultrasound applications to mix, disperse, and dissolve organic materials from a given matrix.

STANDARD ANALYSIS - an analytical determination made with known quantities of target compounds; used to determine response factors.

STORAGE BLANK - reagent water (40.0 ml aliquot) stored with samples. It is analyzed on a weekly basis, and is used to determine the level of contamination acquired during storage.

STOCK SOLUTION - a standard solution which can be diluted to derive other standards.

SULFUR BLANK - a modified method blank that is prepared only when some of the samples in a batch are subjected to sulfur cleanup. It is used to determine the level of contamination associated with the sulfur cleanup procedure. When any of the samples are subjected to sulfur cleanup, then the method blank serves this purpose. When none of the samples are subjected to sulfur cleanup, no sulfur blank is required.

SURROGATES (Surrogate Standard) - for semivolatiles, volatiles and pesticides/Aroclors, compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental media.

SUSPENDED - those elements which are retained by a 0.45 um membrane filter.

SYSTEM MONITORING COMPOUNDS - compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard for volatile analysis, and used to evaluate the performance of the entire purge and trap-gas chromatograph-mass spectrometer system. These compounds are brominated or deuterated compounds not expected to be detected in environmental media.

TARGET COMPOUND LIST (TCL) - a list of compounds designated by the Statement of Work (Exhibit C) for analysis.

TENTATIVELY IDENTIFIED COMPOUNDS (TIC) - compounds detected in samples that are not target compounds, internal standards, system monitoring compounds, or surrogates. Up to 30 peaks (those greater than 10% of peak areas or heights of nearest internal standards) are subjected to mass spectral library searches for tentative identification.

TIME - when required to record time on any deliverable item, time shall be expressed as Military Time, i.e., a 24-hour clock.

TOTAL METALS - analyte elements which have been digested prior to analysis.

TWELVE-HOUR TIME PERIOD - The twelve (12) hour time period for GC/MS system instrument performance check, standards calibration (initial or continuing calibration), and method blank analysis begins at the moment of injection of the DFTPP or BFB analysis that the laboratory submits as documentation of instrument performance. The time period ends after 12 hours have elapsed according to the system clock. For pesticide/Aroclor analyses performed by GC/EC, the twelve hour time period in the analytical sequence begins at the moment of injection of the instrument blank that precedes sample analyses, and ends after twelve hours have elapsed according to the system clock.

VOLATILE COMPOUNDS - compounds amenable to analysis by the purge and trap technique. Used synonymously with purgeable compounds.

WET WEIGHT - the weight of a sample aliquot including moisture (undried).

WIDE BORE CAPILLARY COLUMN - a gas chromatographic column with an internal diameter (ID) that is greater than 0.32 mm. Columns with lesser diameters are classified as narrow bore capillaries.

10% FREQUENCY - a frequency specification during an analytical sequence allowing for no more than 10 analytical samples between required calibration verification measurements, as specified by the contract Statement of Work.

ATTACHMENT B

OF THE

QUALITY ASSURANCE PROJECT PLAN

REFINED METALS CORPORATION SITE

STANDARD OPERATING PROCEDURES

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS FOR ANALYSIS BY FLAA OR ICP SPECTROSCOPY

METHOD 3010A

APPROVALS:

Inorganic Manager: _____ Date: 11/24/98
Douglas E. Kriscunas

QA Manager: _____ Date: 11/24/98
Rick D. Wilburn

Laboratory Manager: _____ Date: 11/24/98
Douglas E. Kriscunas

Procedure Number: GR-01-121
Revision Number: 3.0

Date Initiated: 11/17/96
Effective Date: 8/4/98

Date Revised: 11/24/98
Pages Revised: All

By: Marge Scott

Total Number of Pages: 9

1.0 SCOPE AND APPLICATION

1.1 This method is an acid digestion procedure used to prepare aqueous samples for analysis by flame atomic absorption (FLAA) or by inductively coupled plasma (ICP).

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 1, July, 1992, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA and ICP Spectroscopy".

3.0 SUMMARY OF PROCEDURE

3.1 A representative sample is refluxed in nitric acid until the solution is light in color, or the color of the solution remains stable. The digestate is then refluxed with hydrochloric acid and diluted to a predetermined volume.

3.2 This method has been modified. The initial sample volume has been reduced from 100 ml to 25 ml. The acid amounts have been reduced proportionally to the sample size reduction.

4.0 PARAMETER OR COMPOUND LIST

FLAA or ICP

Aluminum	Magnesium
*Arsenic	Manganese
Barium	Molybdenum
Beryllium	Nickel
Boron	Potassium
Cadmium	*Selenium
Calcium	Silicon
Chromium	Silver
Cobalt	Sodium
Copper	Strontium
Iron	Thallium
Lead	Vanadium
Lithium	Zinc
Tin	
Titanium	

Approved By: _____ **QA Manager**

Approved By: _____ 11/24/98 **Area Manager**

*Analysis by ICP.

5.0 REFERENCED SOPs

5.1 None Referenced.

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Silver precipitation can be avoided by adding an excess of HCl.

6.2 All samples must be at room temperature before the analysis is started.

7.0 SAFETY PRECAUTIONS

7.1 See the "Laboratory Safety Manual" for routine precautions. Gloves and safety glasses must be worn at all times when handling acids. A plastic apron is also recommended. Nitric and hydrochloric acid fumes can burn and therefore respirators are available for use. Rinse all pipet tips, beakers, etc. prior to disposal.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 To ensure enough sample for QC and/or repeats collect at least 500 ml of material to be tested. Preserve to pH <2.0 with trace metal nitric acid.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Eppendorf Pipettor (capable of delivering 250 ul).

9.2 Adjustable pump-type dispenser capable of dispensing 0.75 ml of reagent. This pump is for the concentrated HNO_3.

9.3 Adjustable pump-type dispenser capable of dispensing 2.5 ml of reagent. This pump is for the 1:1 HCl.

9.4 Corning hot Plates (setting #4). Model #PC-500.

9.5 Griffin beakers, 150 ml capacity minimum.

9.6 Ribbed watchglasses.

Approved By: _____ QA Manager

Approved By: _____ 11/24/98 Area Manager

9.7 Non-ribbed watchglasses.

9.8 Class A volumetric flasks.

9.9 Whatman 41 filter paper.

9.10 Plastic funnels.

9.11 Plastic specimen containers.

9.12 Class A Volumetric pipets.

9.13 Graduated cylinder (50 ml).

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Calibrate all pipettors once per week.

10.2 Daily clean all hot plates and counters.

11.0 CHEMICALS AND REAGENTS

11.1 Distilled deionized water (ASTM Type II).

11.2 Concentrated nitric acid, trace metal grade (HNO_3).

11.3 Concentrated hydrochloric acid, trace metal grade (HCl).

11.4 Milli-Q water (ASTM Type I).

11.5 1:1 HNO_3 for cleaning filters. Into 500 ml ASTM Type II water, place 500 ml concentrated HNO_3. Always use a suitable container capable of withstanding the heat generated by the exothermic dilution of the acid.. Always add acid to water (failure to do so could result in a violent explosion of acid as the water boils).

11.6 1:1 HCl. Into 500 ml ASTM Type II water, place 500 ml concentrated HCl. Always use a suitable container capable of withstanding the heat generated by the exothermic dilution of the acid.. Always add acid to water (failure to do so could result in a violent explosion of acid as the water boils).

12.0 STANDARDS PREPARATION

Approved By: _____ QA Manager

Approved By: _____ 11/24/98 Area Manager

12.1 FLAA/ICP spiking solutions (see Appendix A).

12.2 GFAA spiking solutions. 10,000 ppb single-element furnace standards are used for this procedure. Please refer to Appendix B for standard preparation.

13.0 DETAILED PROCEDURE

13.1 Run a pretreatment benchsheet to determine analyses needed (see Appendix F).

13.2 Rinse 25 ml pipets with ASTM Type II water.

13.3 Label a 150 ml griffin beaker with the sample number, client name, and any dilution information that is pertinent.

13.4 Rinse the pipette with the sample, discarding the rinsate. Transfer a 25-ml aliquot of the well-mixed sample into the 150 ml griffin beaker

13.5 We must perform 5% matrix spikes and matrix spike duplicates. If this sample has been designated as needing matrix QC (MS/MSD), then repeat steps 13.3 and 13.4 with two more aliquots of the sample. Label the first aliquot's beaker with "SPK", and the second one as "MSD". **Note:** Shake the sample prior to taking each aliquot.

13.6 Repeat steps 13.2 through 13.5 until there are 20 samples in the batch, or until there are no more samples to be prepared.

13.7 Label 1 beaker as MPB (method preparation blank) and 1 beaker as LFB (laboratory fortified blank). Add 25 ml of ASTM Type II water to each of these beakers using a 50 ml graduated cylinder.

13.8 Spiking FLAA/ICP. Using an Eppendorf pipette, spike the following solutions into each beaker labeled with "SPK", "MSD", or "LFB":

SSW/ 1.0
SSW/SSS 2.0

13.9 In a hood, add 0.75 ml of trace metal grade HNO_3. Cover with a ribbed watchglass and place on a hot plate to evaporate. Adjust the temperature on the hotplate so that a beaker of water in the middle of the hotplate reaches 95°C. Do not allow samples to boil.

13.10 Evaporate the samples to a volume of less than 5 ml. Do not allow samples to boil. Do not allow the samples to go dry. If a sample evaporates to dryness, the sample must be discarded and reprepped in another batch.

Approved By: _____ **Approved By:** 11/24/98
QA Manager **Area Manager**

13.11 Cool the samples.

13.12 Add another 0.75 ml portion of HNO_3 to the samples. Cover the samples with a non-ribbed watchglass and return to the hotplate. Increase the temperature of the hotplate so that a gentle reflux action occurs. Do not let the samples boil. Do not let the samples go to dryness. If a sample evaporates to dryness, the sample must be discarded and reprepped in another batch.

13.13 Continue heating the samples, adding additional 0.75 ml increments of HNO_3 as necessary until the digestion is complete, which will be indicated by the digestate appearing light in color, or not changing in color with additional refluxing. Uncover the beakers, or use a ribbed watchglass, and evaporate to about 3 ml. Do not let any portion of the beaker go dry.

13.14 Cool the samples.

13.15 Add 2.5 ml of 1:1 HCl to the samples. Cover the beakers and reflux for an additional 15 minutes to dissolve any precipitate that may have formed during the digestion.

13.16 Rinse enough 25 ml volumetric flasks for all of the beakers in the digestion batch with ASTM Type II water 3 times.

13.17 For each sample, rinse down the sides of the beaker and the watchglass into the beaker. If there is a significant amount of particulate matter left in the beaker, then either filtration or centrifugation may be employed to remove the substances that could prevent nebulization of the samples.

13.17.1 If the amount of particulate matter will not cause analysis problems, then simply pour the contents of the beaker into a 50 ml volumetric flask. Rinse the beaker walls and bottom with ASTM Type II water into the volumetric flask. Repeat the rinsing 2 more times, collecting each rinse in the volumetric flask. Dilute the flask to volume with ASTM Type II water and mix well. Go to Step 13.18.

13.17.2 If the particulate matter will cause analytical problems, the sample may be centrifuged. Pour the contents of the beaker into a volumetric flask. Rinse the beaker walls and bottom with ASTM Type II water into the volumetric flask. Repeat the rinsing 2 more times, collecting each rinse in the volumetric flask. Dilute the flask to volume with ASTM Type II water and mix well. Pour the contents of the flask into a 50 ml centrifuge tube. Centrifuge the sample until the sediment has settled into the bottom of the tube (see the centrifuge operating manual for loading and operation of the centrifuge). Go to Step 13.18.

13.17.3 If centrifugation is not possible, or does not work, then the sample may be filtered. Prepare a plastic funnel with Whatman 41 filter paper. Rinse the funnel and filter paper with dilute HNO_3, followed by several rinsings of ASTM Type II water. Discard the rinsate. Place the funnel and filter paper in a 50 ml volumetric flask. Pour the contents of the beaker into the

funnel. Rinse the beaker walls and bottom with ASTM Type II water into the funnel. Repeat the rinsing 2 more times, collecting each rinse in the funnel. Rinse the filter paper and funnel with several rinsings of ASTM Type II water Dilute the flask to volume with ASTM Type II water and mix well. Go to Step 13.18

13.18 Pour the contents of the volumetric flask (or centrifuge tube) into a plastic sample cup labeled with the client name, LIMS samples number, and any digestion dilution information. Place the empty volumetric flasks and beakers in a plastic tub dedicated to dirty glassware.

13.19 Fill out the LIMS pretreatment benchsheet and enter the information into LIMS (see Appendix C). Place the batch number on the pretreatment benchsheet.

13.20 Place the lid on the sample cup and set aside. When all of the samples have been placed in cups, place the batch number of the digestion (see step 13.19) on top of each sample cup in the digestion batch. Place the samples in the appropriate vented cabinet in the metals analysis room.

13.21 Completely fill out the metals digestion logbook and analyst notebook (see Appendix D).

13.22 Clean up the area of any spilled material or debris.

14.0 REPORTING AND DELIVERABLES

14.1 All samples that require a digestion must be pretreated on LIMS after digestion. A group of samples digested together using the same digestion method are given a LIMS batch number. This batch number is used by the analysts when analyzing the samples in the metals lab to identify groups of samples. The batch number will also be used in data entry to identify the MPB and LFB for a group of samples. See metals laboratory SOP Appendix C for a detail step by step procedure for this process.

15.0 QUALITY ASSURANCE

15.1 One MPB is carried through the entire process to monitor contamination.

15.2 One LFB is carried through the entire process to monitor method accuracy.

15.3 5% matrix spikes/matrix spike duplicates are performed to determine matrix accuracy and precision.

15.4 If a sample is evaporated to dryness, some metals may be volatilized. If any portion of the bottom of the sample container is allowed to go dry, then the sample must be re-pretreated in another digestion batch.

15.5 Samples must not be allowed to boil. If a sample begins to boil, immediately remove it from the heat and turn the temperature down on the hotplate. Excessive boiling of samples will cause loss of analyte. Samples that have been boiled excessively must be repeated in another digestion batch.

Approved By: _____ QA Manager

Approved By: 11/24/98 Area Manager

15.6 Ensure that the spiking solution being used is within the expiration date. If the standard has expired, do not use the solution. Find another spiking solution that has not expired.

16.0 ANALYST CERTIFICATION/METHOD VALIDATION

16.1 Before the analysis of any actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running a one time analyst certification. While the analyst certification is not instrument/apparatus dependent, this certification is required on every instrument/apparatus that will be running samples to demonstrate instrument ability to generate acceptable accuracy and precision.

16.2 Prepare a quality control check sample spiking standard at a level which will give concentrations suitable for this procedure (a concentration when analyze will fall within 30 – 70 % range of the analytical curve).

16.3 Digest all four check samples following the procedures outlined in this SOP.

16.4 Submit samples to the metals laboratory for the appropriate analysis.

16.5 Calculate the average recovery (x) and the standard deviation of the recovery (s) in the applicable units for each analyte using the four results.

16.5.1 For each analyte x must be in the range 70-130% and s must be less than or equal to 20%. If s and x for all analytes meet the acceptance criteria, the analyst certification is considered acceptable. The analyst and the system are now authorized to run samples by this method. If any individual s exceeds the precision limit or any individual x falls outside the range for accuracy, then the system performance is unacceptable for that analyte.

16.5.2 When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst must proceed according to 16.4.

16.6 Locate and correct the source of the problem and repeat the test for all analytes that failed to meet the criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds beginning with Section 16.1. Samples may not be analyzed by any analyst or on any instrument until the analyst certification has been successfully completed. Copies of the successful analyst certifications/method validation spreadsheet and raw data should be given to the Quality Assurance Manager.

17.0 REFERENCES

17.1 Instruction manuals for pipettors.

Approved By: _____ QA Manager

Approved By: _____ 11/24/98 Area Manager

17.2 SOP on pipet calibration and monitoring.

17.3 USEPA SW846, Method 3010A Revision 1, July 1992

18.0 ATTACHMENTS/APPENDICES

18.1 Example Benchsheets.

Approved By: _____ QA Manager

Approved By: 11/24/98 Area Manager

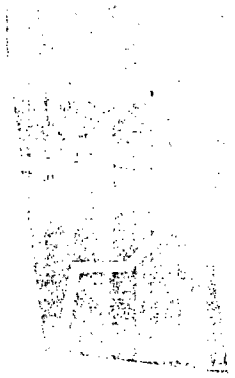
Method: D/FLIC/3010/SPL USEPA-3010A
Test: 903.1-1.3

Date run: 11-5-98
Analyst: MSS
Hours:
Stock Std: 135041

Batch# 135841

Lab Due... Client... Submittal	COC	QC	Bottles	Sample Parameters C F H R		Digested?
09-NOV 33006- 20		LV1	1@16	0 0 0 0	210216 ZnP	✓
09-NOV 33006- 20		LV1	1@16	0 0 0 0	210217 ZnP	✓

DWJ 11-06-98



02-NOV-98 METALS PRETREATMENT BENCHSHEET PAGE 1

Test #: 903.01- 1.03
Parameter: METALS PRETREAT-SPLP/ICP
Method: D/FLIC/3010/SPL
Ref. cit.: USEPA-3010A

Est anal hrs: 1
Act anal hrs:
Stock std #: 53W1446

Owner: MSS
Date run: 11-5-98
Supervisor:
QC Batch #: 13584

Comments: _____

Client.....

Submittal Location	Sample	COC	QC	Anl.	Date Treated	Initial wt/vol	Final wt/vol	Conc. Spiking Solution	SPK STK #	Volume Spiked	Final Spike Conc.	Qu
[redacted] 33006- 20 Overflow P	210216		LV1	MSS	11-5-98	25ml	25ml		53W1446 W152.57	+250		
[redacted] 33006- 20 Overflow P	210217		LV1	↓	↓	↓	↓					
MPB:	1											
LFB: Stk	~											
SPK1: Stk _____ Smp	210216								53W1446 W152.57	+250		
SPK2: Stk _____ Smp	_____											
DUP: _____ Smp	_____											

TRIMATRIX LABORATORIES, INC.

15m7 J66t15m7 J66t

1998-NOV-06 08:12 BATCH DETAIL REPORT -- METALS LABORATORY ANALYTICAL BATCH # 135841 PAGE 1 OF 1

Batch #	Opened	Owner	Seq	Complete	Control	Common name	Application	Ref cit	Qty samp	ICB	ICV	LCS	CCB	CCV
135841	05-NOV-98	MSS	2	YES	YES	D/FLIC/3010/WWT	WW	USEPA-200.2 MOD	0	0	0	0	0	0

	Seq	Sur-seq	QC bat	Run date	Anal	Sample	Appli	Det lim	Init conc	Final conc	Spike	Parameter	Type	%	LCL	UCL	QC	E	D	Ref cit
v	1.000	-		11/05/98	MSS	210216	SPLP					METALS PRETREAT-SPLP/ICP	REG-	1						USEPA-3010A
v	2.000	-		11/05/98	MSS	210217	SPLP					METALS PRETREAT-SPLP/ICP	REG-	1						USEPA-3010A

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

ACID DIGESTION OF SOLID WASTE, SLUDGES, AND SOILS FOR FLAME ATOMIC ABSORPTION, INDUCTIVELY COUPLED PLASMA, OR FURNACE ATOMIC ABSORPTION

USEPA METHOD 3050B, MODIFIED

APPROVALS:

Inorganic Manager: _____ Date: 11/24/98
Douglas E. Kriscunas

QA Manager: _____ Date: 11/24/98
Rick D. Wilburn

Laboratory Manager: _____ Date: 11/24/98
Douglas E. Kriscunas

Procedure Number: GR-01-103
Revision Number: 3.0

Date Initiated: 2/1/96
Effective Date: 11/24/98

Date Revised: 11/24/98
Pages Revised: All

By: Marge Scott

Total Number of Pages: 9

1.0 SCOPE AND APPLICATION

1.1 This method is an acid digestion procedure used to prepare sludges, solid waste, and soil samples for analysis by flame atomic absorption (FLAA), inductively coupled plasma (ICP), or by graphite furnace atomic absorption (GFAA) as indicated by Section 2.0.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 2, December, 1996, "Acid Digestion of Sediments, Sludges, and Soils".

3.0 SUMMARY OF PROCEDURE

3.1 A representative sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with hydrochloric acid if the digestate is to be analyzed by FLAA or ICP. If the digestate is to be analyzed for GFAA, then the sample is refluxed with nitric acid. The final digestate is then filtered and diluted to a predetermined volume.

4.0 PARAMETER OR COMPOUND LIST

FLAA or ICP		GFAA
Aluminum	Molybdenum	Arsenic
Arsenic [1,2]	Nickel	Beryllium
Barium	Osmium	Cadmium
Beryllium	Phosphorus [1,2]	Chromium
Boron [1,2]	Potassium	Cobalt
Cadmium	Selenium [1,2]	Iron
Calcium	Silicon [1,2]	Lead
Chromium	Silver	Molybdenum
Cobalt	Sodium	Selenium
Copper	Strontium [1,2]	Silver
Iron	Thallium	Thallium
Lead	Tin [1,2]	Vanadium
Lithium [1,2]	Titanium [1,2]	
Magnesium	Vanadium	
Manganese	Zinc	

1) This analyte is not covered for digestion in SW-846. Its inclusion in this procedure is due to clients' request for this metal.
2) This metal may only be analyzed by ICP.

5.0 REFERENCED SOPs

5.1 None Referenced.

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Silver precipitation can be avoided by adding an excess of HCl.

6.2 This digestion may not be applicable to all types of solid waste material due to insolubility in the acids and/or water. If the sample superheats during the digestion procedure, an alternative method may have to be used (such as USEPA 3051).

6.3 This digestion will not totally dissolve most matrices. If a total dissolution procedure is required for the sample, the analyst should seek the advice of the metals lab supervisor.

6.4 Contamination is always a major consideration in the analysis of metals. Make sure that the preparation area is free of dust, dirt, etc. before beginning this procedure.

7.0 SAFETY PRECAUTIONS

7.1 The analyst must comply with all standard operating procedures for health and safety as outlined in the "TriMatrix Laboratory Safety Manual".

7.2 Concentrated acids are used in the preparation of samples for analysis. Gloves and safety glasses must be worn at all times when handling concentrated acids. Gloves must also be worn when handling digested samples.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 To ensure enough sample for QC and/or repeats collect at least 30 grams of material to be tested. Sample preservation is not required. Samples must be digested and analyzed within 180 days of collection.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Eppendorf Pipettor (capable of delivering 500 ul).

9.2 Adjustable pump-type dispenser capable of dispensing 2.5 ml of reagent. This pump is for the concentrated HNO_3.

9.3 Adjustable pump-type dispenser capable of dispensing 5.0 ml of reagent. Concentrated HCl.

9.4 Corning hot Plates . Model #PC-500.

9.5 Griffin beakers (identified for 3050-soils).

9.6 Ribbed watch glasses.

9.7 Class A volumetric flasks.

9.8 Whatman 41 filter paper (or equivalent).

9.9 Plastic funnels.

9.10 Plastic specimen containers.

9.11 Balance (capable of weighing to 0.01 grams).

9.13 Spatulas.

9.14 Alcohol thermometers capable of reading 95*C.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Calibrate all pipettors once per week.

10.2 Clean hot plates and counters daily.

11.0 CHEMICALS AND REAGENTS

11.1 Deionized/Distilled water (ASTM Type I).

11.2 Concentrated nitric acid, trace metal grade (HNO_3).

11.3 Concentrated hydrochloric acid, trace metal grade (HCl).

11.4 Hydrogen peroxide (30%) (H_2O_2).

11.4 Milli-Q water (ASTM Type I).

11.5 1:1 HNO_3 for cleaning filters. Into 500 ml ASTM Type II water, add 500 ml concentrated HNO_3. Always use a suitable container capable of withstanding the heat generated by the exothermic dilution of

the acid. Always add acid to water (failure to do so could result in a violent explosion of acid as the water boils).

12.0 STANDARDS PREPARATION

12.1 FLAA/ICP spiking solutions (see Appendix A).

12.2 GFAA spiking solutions. 10,000 ppb single-element furnace standards are used for this procedure. Please refer to Appendix B for standard preparation.

13.0 DETAILED PROCEDURE

13.1 Determine whether samples need to be digested for ICP/FLAA or GFAA.

13.2 Run a pretreatment benchsheet to determine analyses needed (see Appendix F).

12.3 Dry and grind the samples (see Appendix M).

13.4 Label a 150 ml griffin beaker with the sample number, client name, and any dilution factor.

13.5 We must perform 5% matrix spikes and matrix spike duplicates. If this sample has been designated as needing matrix QC (MS/MSD), then repeat step 13.4 with two more aliquots of the sample. Label the first aliquot's beaker with "SPK", and the second one as "MSD".

13.6 Repeat steps 13.4 through 13.5 until there are 20 samples in the batch, or until there are no more samples to be prepared.

13.7 Label 1 beaker as MPB (method preparation blank) and 1 beaker as LFB (laboratory fortified blank). Set aside for now.

13.8 Weigh out the samples.

13.8.1 For FLAA/ICP, weigh 2.50 grams of each remaining sample into 150 ml soil beakers. Record the weight to the nearest 0.01 g. If this sample has been designated for matrix QC, then weigh approximately 10 grams of the mixed sample into a weigh boat and mix again. From the weigh boat, weigh 2.50 g into each of 3-150 ml soil beakers. Record the exact weight to the nearest 0.01 g.

13.8.2 For GFAA, weigh 0.40 grams of each remaining sample into 150 ml soil beakers. Record the weight to the nearest 0.01 g. If this sample has been designated for matrix QC, then weigh approximately 5 grams of the mixed sample into a weigh boat and mix again. From the weigh boat, weigh 0.40 g into each of 3-150 ml soil beakers. Record the exact weight to the nearest 0.01 g.

13.9 Repeat step 13.8 until there are 20 samples in the batch or until there are no more samples to be prepared.

13.10 For ICP/FLAA, add 10 ml of ASTM Type I water to all the beakers, including the MPB and LFB. For GFAA, add 10 ml of ASTM Type I water to all the beakers, including the MPB and LFB.

13.11 All of the samples labeled as SPK, MSD, or LFB must be spiked appropriately before digestion.

13.11.1 For ICP/FLAA, see Appendix A. The final solution volume will be 50 ml.

13.11.2 For GFAA, see Appendix B. The final volume will be 50 ml.

13.12 Add the nitric acid (HNO_3)

13.12.1 For ICP/FLAA, add 2.5 ml of trace metal HNO_3, cover with a ribbed watch glass. Heat the samples to 95°C and reflux for 15 minutes. Do not allow samples to boil or go dry.

13.12.2 For GFAA, add 2.5 ml of trace metal HNO_3, cover with a ribbed watch glass. Heat the samples to 95°C and reflux for 15 minutes. Do not allow samples to boil or go dry.

13.13 Allow the sample to cool. Repeat Step 13.12, refluxing for 30 minutes this time. Do not allow samples to boil or go dry.

13.14 Repeat Step 13.13 to ensure complete oxidation of the sample.

13.15 Reflux the sampled until the volume is reduced to approximately 5 ml. Do not allow samples to boil or go dry.

13.16 If the samples are being prepared for ICP/FLAA, cool, add 2 ml ASTM Type II Water and 10 ml of 30% H_2O_2 very slowly to every beaker in 1 ml increments. If the samples are being prepared for GFAA, cool, add 2 ml ASTM Type II H_2O and 5 ml of 30 H_2O_2 very slowly to every beaker in 1 ml increments. Be sure that the reaction does not cause the sample to boil out of the beaker! Cover the beaker with a watch glass and return the beaker to the hotplate. Warm on a hot plate until the reaction stops. Cool. NOTE: This step may result in a vigorous reaction due to the peroxide. Be sure that the sample does not effervesce over the side of the beaker. If any of the sample is spilled due to this step, the sample must be discarded and reprepped in another digestion batch.

13.17 Continue to add peroxide in 1 ml aliquots with warming until the samples do not change in appearance, the effervescence is minimal, or until 10 ml of peroxide has been added for ICP/FLAA. For GFAA until 5 ml of peroxide has been added.

13.18 Cool the samples.

13.19 If the samples are being prepared for ICP/FLAA, then add 5 ml concentrated trace metal HCl. Cover the beaker and warm for 15 minutes or volume = 10 ml on the hotplate. Cool. Go to Step 13.21.

13.20 If the samples are being prepared for GFAA, then cover the beaker and reflux until the volume has been reduced to about 5 ml. Cool. Go to Step 13.21.

13.21 Rinse 50 ml volumetric flasks with ASTM Type II water 3 times.

13.22 Rinse watch glass into the beaker with ASTM Type II water. Pour the samples in to 50 ml volumetric flasks. Rinse the contents of the beaker into the flask with ASTM Type II water. Rinse the beaker several times with ASTM Type II water. Dilute the samples, LFB, and MPB to 50 ml with ASTM Type II water.

13.23 The samples may now be allowed to settled, centrifuged, or filtered.

13.23.1 Filtration - Rinse Whatman 41 filter paper which has been inserted into a funnel several times with 1:1 HNO_3 followed by several rinsings with ASTM Type II water. Pour the sample through the filter and collect the filtrate into a centrifuge tube or plastic cup. NOTE: If any of the samples within the batch are filtered, all of the samples within the batch must be filtered.

13.24 Label all of the sample containers with the client name, sample number, dilution information, and any other information that may be useful during the analysis. Place the LIMS batch number on the top of the container.

13.25 Pretreat on LIMS to get a batch # (see Appendix C).

13.26 Place samples in appropriate cabinet in metals instrument lab.

13.27 Fill out the pretreatment and lab notebooks (see Appendix D).

14.0 REPORTING AND DELIVERABLES

14.1 All samples that require a digestion must be pretreated on LIMS after digestion. A group of samples digested together using the same digestion method are given a LIMS batch number. This batch number is used by the analysts when analyzing the samples in the metals lab to identify groups of samples. The batch number will also be used in data entry to identify the MPB and LFB for a group of samples. See metals laboratory SOP Appendix C for a detail step by step procedure for this process.

15.0 QUALITY ASSURANCE

Approved By: _____ 11/24/98 (QA Manager) Approved By: _____ 11/24/98 (Area Manager)

15.1 An MPB is carried through the entire process to monitor contamination. One MPB is required for each digestion batch (up to 20 samples).

15.2 An LFB is carried through the entire process to monitor digestion and spiking accuracy. One LFB is required for each digestion batch (up to 20 samples).

15.3 5% matrix spikes must be performed to determine accuracy and check for interferences.

15.4 5% matrix spike duplicates must be performed to determine precision and check for interferences.

15.5 Monitor and document all digestion temperatures as specified section 14.8.

16.0 ANALYST CERTIFICATION/METHOD VALIDATION

16.1 Before the analysis of any actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running a one time analyst certification. While the analyst certification is not instrument/apparatus dependent, this certification is required on every instrument/apparatus that will be running samples to demonstrate instrument ability to generate acceptable accuracy and precision.

16.2 Prepare a quality control check sample spiking standard at a level which will give concentrations suitable for this procedure (a concentration when analyze will fall within 30 – 70 % range of the analytical curve).

16.3 Digest all four check samples following the procedures outlined in this SOP.

16.4 Submit samples to the metals laboratory for the appropriate analysis.

16.5 Calculate the average recovery (x) and the standard deviation of the recovery (s) in the applicable units for each analyte using the four results.

16.5.1 For each analyte x must be in the range 70-130% and s must be less than or equal to 20%. If s and x for all analytes meet the acceptance criteria, the analyst certification is considered acceptable. The analyst and the system are now authorized to run samples by this method. If any individual s exceeds the precision limit or any individual x falls outside the range for accuracy, then the system performance is unacceptable for that analyte.

16.5.2 When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst must proceed according to 16.4.

16.6 Locate and correct the source of the problem and repeat the test for all analytes that failed to meet the criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds beginning with Section 16.1. Samples may not be analyzed by any analyst or on any instrument until the analyst

certification has been successfully completed. Copies of the successful analyst certifications/method validation spreadsheet and raw data should be given to the Quality Assurance Manager.

17.0 REFERENCES

17.1 Instruction manuals for pipettors.

17.2 SOP on pipet calibration and monitoring.

17.3 SW846, November 1986 Third Edition. USEPA Method 3050B Revision 2, Date December 1996.

18.0 ATTACHMENTS/APPENDICES

18.1 Example Benchsheets.

Approved By: RK 12/24/98 Approved By: [illegible] 11/24/98

QA Manager Area Manager

02-NOV-1998 08:11 METALS DIGESTION BENCHSHEET PAGE 1

Method: D/ICP/3050B/SOL USEPA-3050B
Test: 2.2-2.8

Date run: 11-2-98
Analyst: LSS
Hours:
Stock Std: ZLAN

Batch 135712

Lab Due... Client... Submittal	COC	QC	Bottles	C F H R	Sample Parameters	Digested?
09-NOV [redacted] 30620- 18		LV1	1@16	1 0 1 1	210183 AgT AsT CdT SeT	✓
09-NOV [redacted] 30620- 18		LV1	1@16	1 0 1 1	210184 AgT AsT CdT SeT	✓
09-NOV [redacted] 30620- 18		LV1	1@16	1 0 1 1	210185 AgT AsT CdT SeT	✓
09-NOV [redacted] 30620- 18		LV1	1@16	1 0 1 1	210186 AgT AsT CdT SeT	✓
09-NOV [redacted] 30620- 18		LV1	1@16	1 0 1 1	210187 AgT AsT CdT SeT	✓
09-NOV [redacted] 30620- 18		LV1	1@16	1 0 1 1	210188 AgT AsT CdT SeT	✓
09-NOV [redacted] 30620- 18		LV1	1@16	1 0 1 1	210189 AgT AsT CdT SeT	✓
09-NOV [redacted] 30620- 18		LV1	1@16	1 0 1 1	210190 AgT AsT CdT SeT	✓

DWS
11-06-98

003 11810
HW 987191

02-NOV-98

METALS PRETREATMENT BENCHSHEET

Test #: 2.02- 2.08
Parameter: METALS PRETREAT-SOL 3050B
Method: D/ICP/3050B/SOL
Ref. cit.: USEPA-3050B

Est anal hrs: 4
Act anal hrs:
Stock std #: ELAN1

Owner: MSS
Date run: 11-2-98
Supervisor:
QC Batch #: I35712

Comments:

Client.....

Submittal Location	Sample	COC	QC	Anl.	Date Treated	Initial wt/vol	Final wt/vol	Conc. Spiking Solution	SPK STK #	Volume Spiked	Final Spike Conc.	Qu
[redacted]												
30620- 18 SS-17 (0-1	210183		LV1	MSS	11-2-98	0.4g	50ml		ELAN1	500		
[redacted]												
30620- 18 SS-17 (4'-	210184		LV1									
[redacted]												
30620- 18 SS-18 (0'-	210185		LV1									
[redacted]												
30620- 18 SS-18 (7'-	210186		LV1									
[redacted]												
30620- 18 SS-19 (0'-	210187		LV1									
[redacted]												
30620- 18 SS-19 (6'-	210188		LV1									
[redacted]												
30620- 18 SS-20 (0'-	210189		LV1									
[redacted]												
30620- 18 SS-20 (6'-	210190		LV1									

MPB: 1												
LFB: Stk 2												
SPK1: Stk ____ Smp 210185									ELAN1	500		
SPK2: Stk ____ Smp ____												
DUP: ____ Smp ____												

15m7 J66t15m7 J66t

1998-NOV-05 11:57 BATCH DETAIL REPORT -- METALS LABORATORY ANALYTICAL BATCH # 135712 PAGE 1 OF 1

Batch #	Opened	Owner	Seq	Complete	Control	Common name	Application	Ref cit	Qty samp	ICB	ICV	LCS	CCB	CCV
135712	02-NOV-98	MSS	2	YES	YES	D/FLIC/3050/SOL	SOIL	USEPA-3050A	0	0	0	0	0	0

	Seq	Sur-seq	QC bat	Run date	Anal	Sample	Appli	Det lim	Init conc	Final conc	Spike	Parameter	Type	%	LCL	UCL	QC	E	D	Ref cit
v	1.000	-		11/02/98	MSS	210183	SOIL					METALS PRETREAT-SOL 3050B	REG- 1							USEPA-3050B
v	2.000	-		11/02/98	MSS	210184	SOIL					METALS PRETREAT-SOL 3050B	REG- 1							USEPA-3050B
v	3.000	-		11/02/98	MSS	210185	SOIL					METALS PRETREAT-SOL 3050B	REG- 1							USEPA-3050B
v	4.000	-		11/02/98	MSS	210186	SOIL					METALS PRETREAT-SOL 3050B	REG- 1							USEPA-3050B
v	5.000	-		11/02/98	MSS	210187	SOIL					METALS PRETREAT-SOL 3050B	REG- 1							USEPA-3050B
v	6.000	-		11/02/98	MSS	210188	SOIL					METALS PRETREAT-SOL 3050B	REG- 1							USEPA-3050B
v	7.000	-		11/02/98	MSS	210189	SOIL					METALS PRETREAT-SOL 3050B	REG- 1							USEPA-3050B
v	8.000	-		11/02/98	MSS	210190	SOIL					METALS PRETREAT-SOL 3050B	REG- 1							USEPA-3050B

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY

PERKIN ELMER ELAN-6000

USEPA METHODS 6020/200.8

APPROVALS:

Inorganic Manager: _____ Date: 11/23/98
Douglas E. Kriscunas

QA Manager: _____ Date: 11/23/98
Rick D. Wilburn

Laboratory Manager: _____ Date: 11/23/98
Douglas E. Kriscunas

Procedure Number: GR-01-129
Revision Number: 2.1

Date Initiated: 12/4/97
Effective Date: 11/23/98

Date Revised: 11/23/98
Pages Revised: 2,16,29

By: David W. Johnson

Total Number of Pages: 30

1.0 SCOPE AND APPLICATION

1.1 Inductively coupled plasma mass spectrometry (ICP-MS) is a relatively new methodology applicable to a large range of metallic elements in numerous matrices, including, but not limited to: soil, water, drinking water, wastewater, TCLP extracts, EPTox extracts, ASTM extracts, oil, solvents, sludge, air, pure products, and other matrices that may be extracted or dissolved into an acidic aqueous solution. Most matrices require solubilization or digestion prior to analysis.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update III, Revision 0, September, 1994, Inductively Coupled Plasma-Mass Spectrometry.

2.2 *Methods for the Determination of Metals in Environmental Samples*, Supplement I, May 1994, Revision 5.4, EMMC Version, May 1994, "Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry".

3.0 SUMMARY OF PROCEDURE

3.1 Prior to analysis, the sample must be solubilized or digested using an appropriate sample preparation method. See Methods 3005-3050/200.0.

3.2 This method describes the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio frequency inductively coupled plasma. Sample material in solution is introduced by pneumatic nebulization into radio-frequency plasma where energy transfer processes cause desolvation, atomization and ionization. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass-to-charge ratio by a quadrupole are detected by an electron multiplier or Faraday detector and the ion information processed by a data handling system.

3.3 Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Such corrections must include compensation for isobaric elemental interferences and interferences from polyatomic ions derived from the plasma gas, reagents or sample matrix. Instrumental drift as well as suppressions or enhancements of instrument response caused by the sample matrix must be corrected for by the use of internal standards.

4.0 PARAMETER OR COMPOUND LIST

4.1 This method may be used for the analysis of the following metals.

Aluminum	Molybdenum
Antimony	Nickel
Arsenic	Selenium
Barium	Silver

Beryllium	Thallium
Cadmium	Vanadium
Chromium	Zinc
Cobalt	
Copper	
Lead	
Manganese	

5.0 REFERENCED SOPs

5.1 None referenced with regards to the analysis, however all applicable digestion SOPs would be required to pretreat the samples.

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). A data system must be used to correct for these interferences. This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Since commercial ICP-MS instruments nominally provide unit resolution at 10% of the peak height, very high ion currents at adjacent masses can also contribute to ion signals at the mass of interest. Although this type of interference is uncommon, it is not easily corrected, and samples exhibiting a significant problem of this type could require resolution improvement, matrix separation, analysis using another verified and documented isotope, or use of another method. Several interference sources may cause inaccuracies in the determination of trace elements by ICP-MS. These are:

6.1.1 Isobaric elemental interferences - Are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer. All elements determined by this method have, at a minimum, one isotope free of isobaric elemental interference. Of the analytical isotopes recommended for use with this method, only molybdenum-98 (ruthenium) and selenium-82 (krypton) have isobaric elemental interferences. If alternative isotopes having higher natural abundance are selected in order to achieve greater sensitivity, an isobaric interference may occur. All data obtained under such conditions must be corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest. A record of this correction process should be included with the report of the data. It should be noted that such corrections will only be as accurate as the accuracy of the isotope ratio used in the elemental equation for data calculations. Relevant isotope ratios should be established prior to the application of any corrections.

6.1.2 Isobaric polyatomic ion interferences - Are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest, and which cannot be resolved by the mass spectrometer in use. These ions are commonly formed in the plasma or interface system from support gases or sample components. Such interferences must be

recognized, and when they cannot be avoided by the selection of alternative analytical isotopes, appropriate corrections must be made to the data. Equations for the corrections for data should be established at the time of the analytical run sequence as the polyatomic ion interferences will be highly dependent on the sample matrix and chosen instrument conditions. In particular, the common ^{82}Kr interference that affects the determination of both arsenic and selenium, can be greatly reduced with the use of high purity krypton free argon.

6.1.3 Abundance sensitivity - Is a property defining the degree to which the wings of a mass peak contribute to adjacent masses. The abundance sensitivity is affected by ion energy and quadrupole operating pressure. Wing overlap interferences may result when a small ion peak is being measured adjacent to a large one. The potential for these interferences should be recognized and the spectrometer resolution adjusted to minimize them.

6.1.4 Memory interferences - Result when isotopes of elements in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the sampler and skimmer cones, and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element should be estimated prior to analysis. This may be achieved by aspirating a standard containing elements corresponding to ten times the upper end of the linear range for a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of ten of the method detection limit should be noted. Memory interferences may also be assessed within an analytical run by using a minimum of three replicate integrations for data acquisition. If the integrated signal values drop consecutively, the analyst should be alerted to the possibility of a memory effect, and should examine the analyte concentration in the previous sample to identify if this was high. If a memory interference is suspected the sample should be reanalyzed after a long rinse period. In the determination of mercury, which suffers from severe memory effects, the addition of 100 ug/L gold will effectively rinse 5 ug/L mercury in approximately 2 minutes. Higher concentration will require a longer rinse time.

6.2 Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by more than one atom or charge, respectively. Most isobaric interferences that could affect ICP-MS determinations have been identified in the literature. Examples include $ArCl^+$ ions on the ^{75}As signal and MoO^+ ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotope abundances from the literature, the most precise coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<2 percent) counting statistics. Because the ^{35}Cl natural abundance of 75.77 percent is 3.13 times the ^{37}Cl abundance of 24.23 percent, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}Ar^{37}Cl^+$ contribution at m/z 75 is a negligible 0.06 percent of the $^{40}Ar^{35}Cl^+$ signal):

corrected arsenic signal (using natural isotope abundances for coefficient approximations) =

[(m/z 75 signal) - ((3.13)(m/z 77 signal)) + ((2.73)(m/z 82 signal))]

where the final term ((2.73)(m/z 82 signal)) adjusts for any selenium contribution at m/z 77

NOTE: Cadmium values will be biased low by this type of equation when $^{92}ZrO^+$ ions contribute at m/z 108, but use of m/z 111 for Cd is even subject to direct ($^{94}ZrO^+$) additive interferences when Zr is present.

NOTE: As for the arsenic equation above, the coefficients in the Cd equation are ONLY illustrative. The most appropriate coefficients for an instrument can be determined from the ratio of the net isotope signals observed for the standard solution at a concentration providing suitable (<1 percent) counting precision.

6.2.1 The accuracy of these types of equations are based upon the constancy of the OBSERVED isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the "parent" ion have not been found to be reliable, e.g., oxide levels can vary. If a correction for an oxide ion is based upon the ratio of parent-to-oxide ion intensities, the correction must be adjusted for the degree of oxide formation by the use of an appropriate oxide as the interferent. This type of correction has been reported for oxide-ion corrections using ThO^+/Th^+ for the determination of rare earth elements. The use of aerosol desolvation and/or mixed plasmas have been shown to greatly reduce molecular interferences. These techniques can be used provided that method detection limits, accuracy, and precision requirements for analysis can be met.

6.3 Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2%(2,000 mg/L) have been currently recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes. When the intensity level of an internal standard is less than 70 percent or greater than 120 percent of the intensity of the first standard used during calibration, the sample must be reanalyzed after a fivefold (1+4) or greater dilution has been performed.

6.4 Memory interferences can occur when there are large concentration differences between samples or standards, which are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affect the extent of the memory interferences that are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

7.0 SAFETY PRECAUTIONS

7.1 The analyst must comply with all standard operating procedures for health and safety as outlined in the "TriMatrix Laboratory Safety Manual".

7.2 Concentrated acids are used in the preparation of standards and samples for analysis by ICP-MS. Gloves and safety glasses must be worn at all times when handling concentrated acids. Gloves must also be worn when handling digested samples. Please refer to the MSDS for information on these or any other chemicals utilized in this procedure.

7.3 Check the exhaust hood over the instrument to be sure it is operating correctly. If the ventilation system is not working properly, extinguish the plasma if lit and immediately contact the metals lab manager. Under no circumstances should the ICP-MS be used if the exhaust hood is not working.

7.4 UV protective glasses must be worn at all times in the metals laboratory. The plasma emits UV radiation. Avoid looking directly at the plasma without some type of strong UV protection. The instrument uses a very UV resistant material in the viewing port as the analyst may watch the plasma. Do not tamper with this plate. Do not operate the machine without this plate in place. Do not attempt to view the plasma directly or indirectly in any way. Failure to follow this policy may cause very serious and immediate damage to the retina of the eye.

7.5 The ICP-MS emits a strong Rf field. To minimize exposure to this field, Perkin Elmer has included several safety interlocks to prevent direct exposure of the analyst to harmful radiation. Never override any interlock on the ICP-MS. When working on the instrument, always replace all of the Rf shielding using all of the supplied screws. If any safety device has been tampered with, contact the metals lab manager.

7.6 The ICP uses Ar to sustain the plasma and to nebulize sample into the plasma. Although Ar in and of itself is not hazardous or flammable, it may cause suffocation through oxygen deprivation. It is therefore imperative that all sources of Ar be turned off with a valve when not in use. Since Ar is colorless and odorless, if you feel lightheaded, please evacuate the metals lab at once and notify the metals lab manager. Please refer to the MSDS for information on this or any chemicals utilized in this procedure.

7.7 Many of the elements used in the procedure are toxic if ingested. Please refer to the MSDS for information on these or any other chemicals utilized in this procedure.

7.8 No food or drink is allowed in the metals lab. Food or drink may become contaminated with acid or metals and may therefore be hazardous.

7.9 Wash hands before starting work. Chemicals may be present on the skin which may interfere with metals analysis. Wash hands before leaving the metals lab. Chemicals and acids may be on the skin that could eventually be ingested or passed on to a second party through casual contact.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Aqueous samples must be acidified at the time of collection to a pH of <2.

8.2 Solid samples require no preservation for metals.

8.3 All samples may be collected in glass or plastic. If Silicon or Boron is to be analyzed, plastic containers must be used. The acidified sample must never come into contact with any metal as this would cause leaching of the metal into the sample.

8.4 For the analysis of dissolved metals, the sample should be filtered on site and then acidified to a pH of <2.

8.5 Holding times for all metals for ICP analysis is 180 days. In the case of TCLP or similar extracts, the hold time starts at the time of filtration.

8.6 The minimum sample size for this method is 3 ml of aqueous sample per metal to be analyzed. This implies that the sample must be solubilized prior to analysis. A smaller minimum amount may be used if the sample will be diluted at the sacrifice of the detection limit.

8.7 Digested (solubilized) samples and undigested acidified aqueous samples need not be refrigerated. Solid samples should be refrigerated so that other parameters may be performed on them. All samples must be at room temperature prior to analysis.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Inductively coupled plasma mass spectrometer:

9.1.1 Instrument capable of scanning the mass range 5-250 amu with a minimum resolution capability of 1 amu peak width at 5% peak height. Instrument may be fitted with a conventional or extended dynamic range detection system. The instrument should also include data system that will allow corrections for isobaric interferences and the application of the internal standard technique. Use of a mass-flow controller for the nebulizer argon and a peristaltic pump for the sample solution are recommended.

NOTE: If an electron multiplier detector is being used, precautions should be taken, where necessary, to prevent exposure to high ion flux. Otherwise changes in instrument response or damage to the multiplier may result.

9.1.2 Radio-frequency generator compliant with FCC regulations.

9.1.3 Argon gas supply - High purity grade (99.99%). When analysis are conducted frequently, liquid argon is more economical and requires less frequent replacement of tanks than compressed argon in conventional cylinders (Section 4.1.3).

9.1.4 A variable-speed peristaltic pump is required for solution delivery to the nebulizer.

9.1.5 A mass-flow controller on the nebulizer gas supply is required.

9.2 Analytical balance, with the capability of measuring to 0.1 mg for use in weighing solids, for preparing standards, and for determining dissolved solids in digestates or extracts.

9.3 A temperature adjustable hot plate capable of maintaining a temperature of 95° C.

9.4 Recirculator/chiller, Neslab CFT750 or equivalent. Temperature should be maintained at 18° C and pressure should be at 35-60 psi. The recirculator is attached directly to the mass spectrometer to maintain isothermal conditions.

9.5 15 ml clear centrifuge tubes or equivalent. Tubes should stand upright within the autosampler rack without tipping or leaning.

9.6 Eppendorf auto-pipettors capable of delivering 5-1000 ul.

9.7 Class A volumetric flasks, various volumes.

9.8 Class A reusable pipettes, various volumes.

9.9 (optional) An air displacement pipettor capable of delivering volumes ranging from 0.1 to 2500 ul with an assortment of high quality disposable pipette tips.

9.10 Mortar and pestle, ceramic or nonmetallic material.

9.11 Polypropylene sieve, 5-mesh (4 mm opening).

9.12 A gravity convection drying oven with thermostatic control capable of maintaining 105° C ± 5° C.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 All maintenance activities must be recorded in the instrument maintenance, or instrument run logbook. Daily maintenance activities are recorded in the run logbook. All other maintenance activities must be recorded in the instrument maintenance logbook.

10.2 At the beginning of each shift (every 8 hours) the manifold pump tubing must be replaced. This must be recorded in the instrument run logbook.

10.3 There are 2 filters on the ELAN 6000 that must be replaced periodically. The two filters are located on the back of the instrument. This must be noted in the instrument maintenance logbook.

10.4 Inspect the Ar supply when the shift begins. If the liquid level of the Ar falls below the re-order mark, notify the person responsible for ordering gasses or the metals manager. This must be recorded in the instrument run logbook.

10.5 Inspect the water container on the floor alongside the instrument every shift. If the jug is full, properly dispose of the liquid. This must be recorded in the instrument run logbook.

10.6 Visually inspect the torch before lighting the instrument. Do not touch or move the torch. The inspection should be visible only. This must be recorded in the instrument run logbook.

10.6.1 Open the vacuum chamber.

10.6.2 Observe the torch. If the torch is broken or cracked, replace it. See section 10.7 for a discussion on how to disassemble the torch and assembly.

10.6.3 Listen for any obvious leaks in the Ar lines going to the torch and nebulizer. If leaks are heard, investigate them and tighten the fittings to stop the leak. If the leak cannot be found or cannot be stopped, contact the metals lab manager.

10.7 Once every four to eight weeks disassemble the entire torch assembly and inspect all parts for wear. Replace all worn or damaged parts. Record all changes in the instrument maintenance logbook.

10.7.1 Turn off the plasma if it is on by selecting instrument, front panel, and then stop on the plasma box. The pump will stop when the plasma is extinguished.

10.7.2 Turn off the RF Generator by turning off the circuit breaker labeled RF Generator (CB1) on the left side of the instrument.

10.7.3 Loosen the retaining ring and remove the spray chamber assembly.

10.7.4 Open the top left cover of the ELAN 6000. Use a flat blade screw driver on the release mechanism to release the vacuum chamber interlock lever. Grasp the lever and pull in counter clockwise direction to slide the vacuum chamber and interface away from the torch box.

10.7.5 Remove the gas line connections from the ICP torch by loosening the Swagelock fittings. Then slide the fittings and connection tubing up and off of the ICP torch.

10.7.6 To remove the torch assembly, rotate the torch mount 1/8 turn counter clockwise and remove the entire assembly through the right side of the torch box.

10.7.7 With one hand grasping the ICP torch and the other holding onto the lexan adapter, twist and pull back on the adapter to separate the adapter from the torch.

10.7.8 Inspect all O-rings and replace any that are cracked or damaged. Record in maintenance log book.

10.7.9 Remove the Alumina injector. If it is dirty wash it with soap and water, dry, then put in the muffle furnace at 700° C for 20 min.

10.7.10 If the torch is dirty immerse it in an ultrasonic cleaner containing 1% nitric acid. Rinse with deionized water. If the torch cannot be cleaned or is damaged, replace.

10.7.11 After the torch has been cleaned or replaced, follow these steps to reposition and align the ICP torch.

10.7.11.1 Slide the injector/support adapter to the back of the torch.

10.7.11.2 Place the torch through the hole in the right side of the torch box. Rotate the torch assembly 1/8 turn clockwise until the bayonet mount is fully seated.

10.7.11.3 Reattach the gas lines to the ICP torch. Slide both the fittings and connecting tubing onto the torch and tighten the Swagelock fittings several turns.

10.7.11.4 To check the position and measure for the proper alignment of the torch follow steps 10.7.11.5 through 8.

10.7.11.5 Place the torch alignment tool on the torch so that the flange of the alignment tool touches the first turn of the load coil. Loosen the torch locking collar approximately 1 cm and slide the torch so that it is lined up with the outer edge of the torch.

10.7.11.6 Slide the tool out approximately 1/2 inch and, using the lever, move the vacuum chamber interlock into the lock position.

10.7.11.7 Move the vacuum chamber to the closed position to locate the alignment tool within the load coil.

10.7.11.8 Move the chamber to the open position and adjust the vacuum chamber position so the top end of the torch is even with the 5.5 mm cutout depth of the alignment tool.

10.7.11.9 If the torch is not properly lined up, loosen the two locking screws of the vacuum chamber interlock. Adjust the vacuum chamber forwards or backwards until the proper position is achieved.

10.7.11.10 Slide the vacuum chamber and interface toward the torch box. Secure the vacuum chamber interlock and close the top cover.

10.7.11.11 Turn on the RF Generator by turning on the circuit breaker labeled ICP Power (CB1) on the side of the instrument.

10.7.12 Inspect the drain line at the base of the spray chamber. There should be no leaks or cracks in the tubing. If a problem exists, replace the O-ring in the drain cap. This must be recorded in the instrument maintenance logbook.

10.7.13 Inspect the nebulizer for wear. This must be recorded in the instrument run logbook.

10.7.13.1 Salt deposits, if present, should be removed with water. Record any actions in the instrument maintenance logbook.

10.7.13.2 If a cross-flow nebulizer is used, check the tips for wear. Replace the tips if excessive wear is noted. Record any actions in the instrument maintenance logbook.

10.8 Inspect the printing quality of the printer. If the print is hard to read or light in appearance, replace the printer cartridge with the appropriate replacement.

11.0 CHEMICALS AND REAGENTS

11.1 Acids used in the preparation of standards and for sample processing must be reagent grade or better. Redistilled acids may be used if it has been demonstrated that the acid is free from contamination.

11.1.1 Concentrated Nitric acid, trace metal grade (Fisher Cat #A509-212).

11.1.2 Nitric acid, trace metals grade (1:1): Add 500 ml concentrated HNO_3 to 400 ml ASTM Type II water. Cool. Dilute to 1L.

11.2 ASTM Type II water (ASTM D1193). Deionized water is fed into a Barnstead Model FI-STREEM 2 all glass distillation unit. The resulting distillate is immediately placed into a plastic container. Impurities are measured by the Inorganic group at TriMatrix. This is the only water acceptable for use in the Metals lab for dilutions or standard preparation.

11.3 Standard stock solutions are purchased primarily form Inorganic Ventures. All stock solutions are ICP grade single-element solutions at concentrations of 1000 or 10,000 ppm.

11.4 Argon gas supply: Welding grade or better. This is plumbed from a liquid argon tank located outside of the building. Ar is used as the main plasma gas, the auxiliary plasma gas, the nebulizer carrier gas, and the Rf coil coolant.

12.0 STANDARDS PREPARATION

12.1 All standards should be prepared with ASTM Type II water and 2% HNO_3 (for analysis of all samples).

12.2 All primary standards expire one year after receipt or on the date located on the standard bottle, whichever is earlier.

12.3 All working standards and dilutions of working standards for the ICP that are prepared from primary standards expire 3 months after preparation.

12.4 Prepare the 20 ppm working standard ELAN A.

12.4.1 Wash a plastic 100 ml volumetric flask with Citranox and hot water. Put 2 ml of concentrated HNO_3 acid into the volumetric and shake. Rinse the volumetric with ASTM Type II water a minimum of three times.

12.4.2 Place about 10 ml ASTM Type II water into the flask.

12.4.3 Pipette 2 ml HNO_3 into the flask and swirl.

12.4.4 Pipette 2 ml of each of the following standards into the flask, swirling after each addition:

Be, Al, V, Cr, Mn, Co, Ni, Cu, Zn, As, Se, Ag, Cd, Ba, Tl, Pb

Always use a new pipette for each standard and never pipette directly out of the primary standard bottle

12.4.5 Dilute the flask to volume with ASTM Type II water and mix well.

12.4.6 Pour the contents of the flask into a clean bottle and label the bottle to describe its contents.

12.4.7 Update the standard log book. Transfer the stock standard number and preparation date from the book to the standard bottle.

12.5 Prepare the 20 ppm standard ELAN B

12.5.1 Wash a 100 ml plastic volumetric with Citranox and hot water. Put 2 ml of concentrated HNO_3 acid into the volumetric and shake. Rinse the volumetric with ASTM Type II water a minimum of three times.

12.5.2 Place about 10 ml ASTM Type II water into the flask.

12.5.3 Pipette 2 ml HNO_3 into the flask and swirl.

12.5.4 Pipette 2 ml each of the 1000 ppm Sb and Mo stock standards into the flask, swirling after each addition. Always use a new pipette for each standard and never pipette directly out of the primary standard source bottle.

12.5.5 Dilute the flask to volume with ASTM Type II water and mix well.

12.5.6 Pour the contents for the flask into a clean bottle and label the bottle to describe its contents.

12.5.7 Update the standard log book. Transfer the stock standard number and preparation date from the book to the standard bottle.

12.6 Prepare the mixed working standards.

12.6.1 Rinse seven 50 ml, and one 100 ml volumetric flasks with ASTM Type II water a minimum of three times.

12.6.2 Place about 20 ml ASTM Type II water into each volumetric flasks.

12.6.3 Each standard should have an acid concentration of 2%. Pipette 1 ml of concentrated nitric acid into each of the 50 ml volumetric flasks, and 2 ml into the 100 ml volumetric flask to achieve this concentration.

12.6.4 Pipette 25 ul of ELAN A solution and 25 ul ELAN B solution into one of the 50 ml volumetric flasks. Dilute to volume with ASTM Type II water and mix. This is a 10 ppb standard.

12.6.5 Pipette 50 ul of ELAN A solution and 50 ul ELAN B solution into one of the remaining 50 ml volumetric flasks. Dilute to volume with ASTM Type II water and mix . This is a 20 ppb standard.

12.6.6 Pipette 125 ul of ELAN A solution and 125 ul of ELAN B solution into one of the remaining 50 ml volumetric flasks. Dilute to volume with ASTM Type II water and mix. This is a 50 ppb standard.

12.6.7 Pipette 250 ul of ELAN A solution and 250 ul of ELAN B solution into one of the remaining 50 ml volumetric flasks. Dilute to volume with ASTM Type II water and mix. This is a 100 ppb standard.

12.6.8 Pipette 40 ul of the 100 ppb standard into the 100 ml volumetric flask. Dilute to volume with ASTM Type II water and mix. This is a 0.04 ppb standard.

12.6.9 Pipette 100 ul of the 100 ppb standard into one of the remaining 50 ml volumetric flasks. Dilute to volume with ASTM Type II water and mix. This is a 0.20 ppb standard.

12.6.10 Pipette 500 ul of the 100 ppb standard into one of the remaining 50 ml volumetric flask. Dilute to volume with ASTM Type II water and mix. This is a 1.0 ppb standard.

12.6.11 Dilute the acid to volume in the final 50 ml volumetric flask with ATSM Type II water and mix. This is a 0 ppb standard.

12.6.12 Update the standard log book. Transfer the stock standard numbers and preparation date to the standard bottles.

12.7 Prepare the Interference Check Solutions (ICS).

12.7.1 The ICSs are prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interferences and provide an adequate test of any corrections. Chloride in the ICS provides a means to evaluate software corrections for chloride-related interferences such as $^{35}Cl^{16}O^{+}$ on $^{51}V^{+}$ and $^{40}Ar^{35}Cl^{+}$ on $^{75}As^{+}$. Iron is used to demonstrate adequate resolution of the spectrometer for the determination of manganese.

Molybdenum serves to indicate oxide effects on cadmium isotopes. The other components are present to evaluate the ability of the measurement system to correct for various molecular-ion isobaric interferences. The ICS is used to verify that the interference levels are corrected by the data system within quality control limits.

12.7.2 These solutions must be prepared from ultra-pure reagents. They can be obtained commercially or prepared by the following procedure.

12.7.2.1 Mixed ICS solution I is prepared by weighing into individual weigh boats the following chemicals: 1.39g $Al(NO_3)_3 \cdot 9H_2O$, 0.25g $CaCO_3$ (dried at 180°C for 1 hour before weighing), 0.10g Fe, 1.7g MgO, 0.23g Na_2CO_3, and 0.18g K_2CO_3. These are then added to a 100 ml volumetric flask containing approximately 25 ml of ASTM Type II water. Slowly add 4 ml of (1+1)HNO_3. After dissolution is complete, warm the solution to degas. Cool and dilute to volume ml with ASTM Type II water.

12.7.2.2 Mixed ICS solution II is prepared by weighing into individual weigh boats the following chemicals: 0.74g 85% H_3PO_4, 0.64g 96% H_2SO_4, 4.0g 37% HCl, and 1.1g citric acid ($C_6O_7H_8$). These are then added to a 100 ml volumetric flask containing approximately 25 ml of ASTM Type II water. Dilute to volume with ASTM Type II water.

12.7.2.3 Mixed ICS solution III is prepared by adding 1.0 ml each of 100 ug/ml arsenic, cadmium, chromium, cobalt, copper, manganese, nickel, silver, and zinc stock solutions to a 100 ml volumetric flask approximately half full of ASTM Type II water. Add 2.0 ml concentrated HNO_3, and dilute to volume with ASTM Type II water.

12.7.2.4 Working ICS Solutions

12.7.2.4.1 ICS-A is prepared by adding to a 100 ml volumetric flask, 10.0 ml of mixed ICS solution I, 5.0 ml of mixed ICS solution II, and 2.0 ml each of 100 ug/ml titanium and molybdenum stock solutions. Dilute to volume with ASTM Type II water and update the standard logbook. ICS-A must be prepared fresh weekly.

12.7.2.4.2 ICS-AB is prepared by adding to a 100 ml volumetric flask, 10.0 ml of mixed ICS solution I, 5.0 ml of mixed ICS solution II, 2.0 ml of mixed ICS solution III, and 2.0 ml each of 100 ug/ml titanium and molybdenum stock solutions. Dilute to volume with ASTM Type II water. Although ICS-AB must be prepared fresh weekly, the analyst should be aware that the solution may precipitate silver more quickly. Update the standard log book.

12.7.2.5 The following analytes will be analyzed for in the ICS solutions at the stated concentrations:

ICS-A Working Solution

Element	Concentration (ppm)
Al	100
Mo	20

ICS-AB Working Solution

Element	Concentration (ppm)
Al	100
As	0.02
Cr	0.02
Cu	0.02
Mn	0.02
Mo	2.0
Ni	0.02
Se	0.02
Co	0.02
Ag	0.02
Cd	0.02

13.0 SAMPLE PREPARATION

13.1 Soil, sludge, and waste samples must be digested according to method 3050 prior to analysis. Please see the specific SOP for the detailed preparation procedure.

13.2 Samples that have had an extraction performed on them, such as TCLP, SPLP, EPTox, and ASTM, must first be extracted then be digested using method 3010 or 3015. Please see the specific SOP for the detailed preparation procedure.

13.3 Wastewater samples must be prepared using method 3010 or 3015. Please see the specific SOP for the detailed preparation procedure.

13.4 Oil samples may be digested using method 3051. Please see the specific SOP for the detailed preparation procedure.

13.5 Air samples are prepared using the digestion method specified by the method used in the collection of the samples.

13.6 Pure product samples are prepared according to the manufacturer, or according to a method developed by TriMatrix if available. Please see the specific SOP or dissolution procedure provided by the manufacturer or TriMatrix for the detailed preparation procedure.

13.7 Drinking water samples must have the turbidity checked prior to analysis. Any sample with a turbidity greater than 1 NTU must be digested by method 3015 prior to analysis. Any dissolved sample that has a precipitate must be digested by method 3015 prior to analysis.

14.0 CALIBRATION PROCEDURES

14.1 An initial calibration must be run prior to the analysis of any samples. This initial calibration must be performed daily, or once per every 24 hour analytical batch.

14.1.1 The initial calibration consists of an 8 point standard curve for silver and cadmium, and a minimum of a seven point calibration curve for all other analytes. The initial calibration curve consists of the standards prepared in 12.6. The silver and cadmium curve includes the 0.04 ppb standard. This concentration is not included when processing the curve for the other analytes. The 0 ppb is included as a standard in both curves.

14.1.2 The correlation coefficient must be greater than or equal to 0.995 for every analyte of interest. If the coefficient is less than 0.995, contact the metals lab manager for assistance. All masses that could affect data quality should be monitored to determine potential effects from matrix components on the analyte peak.

14.2 Initial Calibration Verification (ICV).

14.2.1 Immediately following the analysis of the curve, the analysis of an ICV is required. The ICV is a re-analysis of the high level 100 ppb standard prepared in 12.6.7, quantitated as a sample. The acceptance window for this QC is 90% to 110% recovery of the true value. If the ICV is not within the control limits, recalibrate the instrument and start again at step 14.2. If the ICV is still out of the control limits, see the metals lab manager.

14.3 Initial Calibration Blank (ICB)

14.3.1 After the analysis of the ICV, the analysis of an ICB is required. The ICB is a re-analysis of the 0 ppb standard prepared in 12.6.11, quantitated as a sample. The absolute value of the ICB reading must be less than the lowest reporting limit required for the analysis. If the ICB is not within the control limits, recalibrate the instrument and start again at step 14.2. If the ICB is still out of the control limits, see the metals lab manager.

14.4 Interference Check Solutions (ICS).

14.4.1 The interference check solutions were prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interferences and provide an adequate test of any corrections. The acceptance window for the interference check solution is 80% to 120%. If either one of the interference check solutions are outside of the control windows stop the analysis remake the out of control solution(s) and reanalyze. If the interference check solution(s) are now within the control windows continue the analysis. If either of the

interference check solutions are still out of the control windows stop the analysis, correct the problem, and restart the analysis from the beginning.

14.5 Laboratory Control Sample (LCS).

14.5.1 A LCS must be analyzed to confirm the validity of the curve. The LCS is prepared from a stock dissimilar to that used to prepare initial calibration curve. The control windows for the LCS 90% to 110% recovery of the true values. If the percent recovery does not fall within the control windows, recalibrate the instrument and start the analysis at step 14.2.

14.6 Contract Required Detection Limit (CRDL)

14.6.1 Analyze a CRDL. This is a standard of known concentration at or near the low end of your calibration curve. There is no acceptance criteria for the CRDL other than it must be detected.

14.7 Analyze up to ten samples. A sample is defined as the average of three replicate readings of a solution that is not a standard. The following solutions would qualify as samples: LCSs, MPBs, LFBs, and spiked samples. The only solutions that are not considered samples are the ICV, ICS, ICB, CCB, CCV, and CCB solutions.

14.8 Continuing Calibration Verification (CCV)

14.8.1 Analyze the CCV. The CCV is a standard solution with a concentration of one half the highest standard solution used for the calibration. The acceptance window for this QC is 90% to 110% recovery of the true value. If the CCV is not within the control limits, recalibrate the instrument and start again at step 13.2. All samples since the last good ICV or CCV must be reanalyzed.

14.9 Continuing Calibration Blank (CCB)

14.9.1 Analyze the CCB. The CCB is the calibration blank and has the same acid concentration as the standards. The absolute value of the CCB reading must be less than the lowest reporting limit required for the analysis. If the CCB is not within the control limits, recalibrate the instrument and start again at step 13.2. All samples since the last good ICB or CCB must be reanalyzed.

14.10 Every twelve hours of instrument operation the ICS solutions must be analyzed to verify the magnitude of elemental and molecular ion isobaric interferences and the adequacy of any corrections. The acceptance window for this QC is 80% to 120% recovery of the true value. If the QC is not within the acceptance window for either ICS, stop the analysis, remake the interference check solution and reanalyze. If the QC is now within the acceptance window continue the analysis. If the QC is still outside the acceptance window stop the analysis, correct the problem, and restart the analysis from the beginning.

14.11 Repeat steps 14.7 through 14.9 until the end of the run.

14.12 The analytical batch must end with the analysis of an acceptable CCV and CCB. Again, the acceptance window for the CCV is 90% to 110% recovery of the true value, and the absolute value of the CCB reading must be less than the lowest reporting limit required for the analysis. If either is not within the control limits, recalibrate the instrument and start again at step 14.2. All samples since the last good CCV or CCB must be reanalyzed.

15.0 ANALYTICAL PROCEDURE

NOTE: The following section assumes that the user is familiar with the ELAN 6000 operating software. If you are unfamiliar or unsure, please consult the instrument manual for additional instruction. This procedure will provide adequate instruction for novices, but is not intended to replace the instrument operating manual.

15.1 Preliminary treatment of all matrices is always necessary because of the complexity of variability of sample matrices. Digestion procedures are presented in sample preparation methods (method 3005-3050/200.0/200.7). A minimum of five internal standards is always used.

15.2 Turn on the instrument.

15.2.1 Turn on the main power if it is not already on.

15.2.2 Turn on the Rf generator power if it is not already on.

15.2.3 Turn on the autosampler power if it is not already on.

15.2.4 Turn on the recirculator power if it is not already on.

15.2.5 Turn on the Ar gas supply if it is not already on.

15.2.6 Turn on the hood above the instrument if it is not already on.

15.3 Boot the computer and ELAN 6000 software.

15.3.1 If the computer is already on the ELAN software is running, skip to step 14.4. If you are unsure of the status of the computer, please see the metals manager.

15.3.2 Turn on the computer, monitor, and printer. If any of the devices fail to turn on, please see the metals manager.

15.3.3 When the computer boots, it will display:
Welcome
Press Ctrl + Alt + Del to log on.

15.3.4 The screen will display:
Welcome
username: Elan user
from: ICP-MS
password: ____
Just press enter do not enter anything as a password.

15.3.5 The screen will display:
Program manager and Elan 6000 (common)
move the arrow to Elan and click on it

15.3.6 The screen will display:
a general Elan 6000 window
move arrow to devices and click on it. This will bring up the pump page.

15.4 Change the peristaltic pump tubing. Currently, three types of tubing are being used:

Black/Black	Sample line
Orange/Green	Internal standard line
Black/White	Waste line

The tubing should be changed for every run since the tension exerted on the tubing by the pump will cause the tubing to stretch. Place the waste line on the bottom so that the pump is pumping the waste away from the ICP. Place the internal standard line in the middle so that the pump is pumping the internal standard toward the ICP. Place the sample line on the top so that the pump is pushing the sample to the ICP. Update the instrument maintenance log to reflect that you change the pump tubing.

15.5 Click on the connect icon box then click on the ---> icon box this should turn the pump on in a counterclockwise direction. If the pump does not turn on. Click on the disconnect icon. Then click on the connect icon. Then click on the ---> icon and the pump should be turning in the counterclockwise direction. If the pump still is not on contact the metals lab manager.

15.6 Move the arrow to the instrument icon and click on it. There should be a picture of the ICP-MS and some of its components on the screen if there is not click on the front panel icon.

15.6.1 On the picture of the ICP-MS. There should be a number of components that are green and the status box should say Ready. Consult the instrument manual as to which components should be green.

15.6.2 If the status is Ready, move the arrow to the plasma icon box and click on start. The instrument will display an ignition icon bar until it has achieved a good plasma.

15.7 Once the plasma has been on for at least one half hour daily tuning of the instrument needs to be done.

15.7.1 Move the arrow to File. Highlight open workspace then click on x,y.wrk. This will align the plasma to the best spot on the sampler and skimmer cones. Place the sample tubing into a 10 ppb tuning solution of Rhodium, Lead, and Magnesium. Click on Analyze Sample and adjust

the x,y knobs to get the highest Rhodium reading. This is typically 250,000 cps to 300,000 cps.

15.7.2 Next you will need to optimize the nebulizer flow and lens voltage. Move the arrow to File and highlight NLP.wrk. Place the sample line in the 10 ppb tuning solution and make sure that nebulizer flow is highlighted. Click on Analyze Sample. The computer will automatically adjust the nebulizer flow between 0.4 and 0.7 L/min. This range can be changed but has been found to be the best. When the instrument is finished go to File and click on Save.

15.7.3 Next highlight Lens Voltage and click on Analyze Sample. The computer will automatically pick the best lens voltage for you. The value is typically from 4 to 6 if it is not fix the problem and start the tuning procedure form 14.7.1. When the instrument is finished go to File and click on Save.

15.7.4 Next you will need to tune the mass spec. over the whole mass range. Go to File click on Open workspace. Click on TriMatrix Int.wrk. Place the internal standard line into the mixed internal standard bottle. Click on Tune Mass Spec. You want to get a value of 0.65 amu or less at 10% peak height. This has been shown by the instrument manufacturer to equal 1 amu peak width at 5% peak height. If the required resolution was not achieved. Manually adjust the DAC and try again, generally increasing it by 30 will decrease the Amu reading by 0.1. When the desired Amu reading has been achieved for all the referenced analytes then go to File and click on Save.

15.7.5 Next do a daily performance to see how the instrument is operating. Go to File and click on Daily.wrk. Place the sample probe into the 10 ppb tuning solution and click on Analyze Sample. The instrument will take a reading of certain masses and doubly charged ions and oxides. All % RSDs should be less than 5% if not repeat the procedure once. If it is in the limits go to 14.7.6. If not stop the tuning procedure. Correct the problem and begin again for 14.7.1.

15.7.6 Next you will need to optimize the ion lens over the full mass range. Go to File and click on Open Workspace. Then Lens Optimization. Put the internal standard lime into the mixed internal standard bottle. Click on Clear Calibration. Then get analyte list, click on Optimize. The computer will automatically adjust the voltage to the ion lens finding the maximum value for each internal standard. When the optimization is done go to File and click on Save.

15.7.7 Once all of the optimization steps are complete you are ready to calibrate and run samples.

15.8 The table below lists the element conditions in use at the time of this writing. Please note that any or all of the conditions listed below may change for an element without prior warning if it has been established that the new conditions are at least equivalent to those in the table.

Approved By: _____ 11/23/98 Approved By: _____ 11/23/98

QA Manager Area Manager

Int Std	Analyte	Mass [amu]	Scan Mode	MCA Channels	Dwell Time per AMU [ms]	Integration Time [ms]	Corrections
Li		6.0151	Peak Hopping	1	100	1000	
	Be	9.0122	Peak Hopping	1	100	1000	
Sc		44.9559	Peak Hopping	1	100	1000	
	V	50.944	Peak Hopping	1	100	1000	C10, Cr
	Cr	52.9407	Peak Hopping	1	100	1000	
	Mn	51.9405	Peak Hopping	1	100	1000	
	Co	58.9332	Peak Hopping	1	100	1000	
	+Ni	59.9332	Peak Hopping	1	100	1000	
	Ni	60.931	Peak Hopping	1	100	1000	
	+Cu	62.9298	Peak Hopping	1	100	1000	
	Cu	64.9278	Peak Hopping	1	100	1000	
	+Zn	65.926	Peak Hopping	1	100	1000	
	Zn	66.9271	Peak Hopping	1	100	1000	
	Zn	67.9249	Peak Hopping	1	100	1000	
	As	74.9216	Peak Hopping	1	100	1000	ArCl, Se
	Se	81.9767	Peak Hopping	1	100	1000	
Ge		88.9054	Peak Hopping	1	100	1000	
	Mo	97.9055	Peak Hopping	1	100	1000	Ru
	+Ag	106.905	Peak Hopping	1	100	1000	
	Ag	108.905	Peak Hopping	1	100	1000	
	+Cd	110.904	Peak Hopping	1	100	1000	Mo, Pd
	Cd	113.904	Peak Hopping	1	100	1000	Sn
In		114.904	Peak Hopping	1	100	1000	Sn
	Sb	120.904	Peak Hopping	1	100	1000	
	+Sb	122.904	Peak Hopping	1	100	1000	Te
	Ba	134.906	Peak Hopping	1	100	1000	
	+Ba	136.905	Peak Hopping	1	100	1000	
Tb		158.925	Peak Hopping	1	100	1000	
	Tl	202.972	Peak Hopping	1	100	1000	
	+Tl	204.975	Peak Hopping	1	100	1000	
	Pb	207.977	Peak Hopping	1	100	1000	Pb, Pb
Bi		208.980	Peak Hopping	1	100	1000	
*	Br	208.980	Peak Hopping	1	0.1	1	
*	Cl	34.9689	Peak Hopping	1	0.1	1	
*	Ca	43.9555	Peak Hopping	1	0.1	1	
*	Mg	23.985	Peak Hopping	1	0.1	1	
*	Na	22.9898	Peak Hopping	1	0.1	1	
*	Fe	56.9354	Peak Hopping	1	0.1	1	
*	K	38.9637	Peak Hopping	1	0.1	1	
*	Ar Cl	76.9283	Peak Hopping	1	0.1	1	
*	Kr	82.9141	Peak Hopping	1	0.1	1	
*	Ru	100.906	Peak Hopping	1	0.1	1	
*	Pd	105.903	Peak Hopping	1	0.1	1	
*	Mo^{O}	107.901	Peak Hopping	1	0.1	1	
*	Sn	117.902	Peak Hopping	1	0.1	1	
*	Te	124.904	Peak Hopping	1	0.1	1	

* Used for reference purposes only
+ Recommended Analytical Mass

Approved By: [signature] 11/23/98 QA Manager
Approved By: [signature] 11/23/98 Area Manager

15.8.1 The Table below lists the elemental equations being used at this time. Please note that any or all of these equations may change without prior warning if it has been established that the new equations are at least equivalent to those below:

Analyte	Equation
Al	$(1.00)(^{27}C)$
Sb	$(1.00)(^{123}C)$
Sb	$(1.00)(^{121}C)$
As	$(1.00)(^{75}C)-(3.127)[(^{77}C)-(0.815)(^{82}C)$
Ba	$(1.00)(^{137}C)$
Ba	$(1.00)(^{135}C)$
Be	$(1.00)(^{9}C)$
Cd	$(1.00)(^{111}C)-(1.073)[(^{108}C)-(0.712)(^{106}C)]$
Cd	$(1.00)(^{114}C)$
Cr	$(1.00)(^{52}C)$
Cr	(1.00)(C)
Co	$(1.00)(^{59}C)$
Cu	$(1.00)(^{63}C)$

15.9 Set up the analysis on paper using a sample identification weight sheet (ID/WT sheet). This sheet has columns where the operator enters the sample description, corresponding autosampler position number, digestion dilution (if any), and any subsequent dilutions performed on the sample. This sheet must be filled out before continuing with the procedure as it is an integral part of the analysis. Please see the group leader or metals lab manager if assistance is required in filling out this sheet.

15.9.1 Enter the method by moving the arrow to File and clicking on it. Then click on open workspace you will have a number of choices, but only choose between 200.8 Trimatrix.wrk, and Trimatrix soil.wrk.

15.9.2 Open a new data set. To do this move the arrow to the data set icon and click on it. Then move the arrow to file and click on it and choose new. You will need to enter a unique data file name. The current data file format is MDDAAAA-X,
where:
M = coded month
DD = two digit date of the month
AAAA = matrix that is running (i.e. soil, wtr, TCLP, WW)
X = Alpha representation of the number of runs that have been done under that matrix for that day (i.e. the first run would be 'a', the 5th run would be 'e').

15.9.3 Enter the sample list as identified on the ID/WT sheet. Please refer to the ELAN 6000 software manual for data entry procedures. When all the information is entered you will need to save the sample file. To do this go to file and click on save as. Use the same data file name here as you used for the data set.

15.10 Begin the analysis.

15.10.1 Make sure that batch is highlighted . Highlight the samples that you want to run and click on the analyze batch icon.

15.10.2 The computer will ask you a series of yes and no questions. If you are starting a new run answer yes to all of the questions. Then the computer will start the run.

15.10.3 A measurement status box will be displayed on the screen at this time. It will tell you what the instrument is doing. It will also give you three options to stop a run at any time click on canal. To stop after the current sample click on stop scanning after current sample, or to skip the sample click on skip scanning of current sample.

15.10.4 If you have stopped a run for any reason and want to continue the analysis from where you left off go to 14.6.1.

15.11 When the analysis is completed, the autosampler will return to position 0 and wait for operator input.

15.12 Turn the raw data, ID/WT sheet, and report to the person who is to review the data for repeats, dilutions, etc.

16.0 CALCULATIONS AND DATA HANDLING

16.1 The concentration of each sample is read directly from the computer printout. Dilution factors should be taken into account in the ID weight file.

16.1.1 Assuming that the instrument did not correct a concentration due to analyst error, the following calculation would be used:

$C_{Final} = C_{Instr} * DF_{Digest} * DF_{Subseq}$,, where:

C_{Final} = final reported concentration of the analyte
C_{Instr} = concentration as read from the instrument data printout
DF_{Digest} = digestion dilution factor
DF_{Subseq} = dilution due subsequent to the digestion dilution

16.2 All samples should be reported to the correct number of significant figures. The significant figure truncation should not be performed until all data calculation have taken place.

16.2.1 Solid samples must be reported in mg/kg.

16.2.1.1 For sample concentrations <100 ppm, report 2 significant figures.

16.2.1.2 For sample concentrations ≥ 100 ppm, report 3 significant figures.

16.2.1.3 For QC always report one additional significant figure.

16.2.2 Aqueous samples must be reported in ug/L.

16.2.2.1 For sample concentrations < 10 ppb, report 1 significant figure.

16.2.2.2 For sample concentrations ≥ 10 ppb and < 1000 ppb, report 2 significant figures.

16.2.2.3 For sample concentrations ≥ 1000 ppb, report 3 significant figures.

16.2.2.4 For QC always report one additional significant figure.

16.2.3 Extracted samples for all metals must be reported in mg/L.

16.2.3.1 For sample concentrations <0.01 ppm, report 1 significant figure.

16.2.3.2 For sample concentrations ≥ 0.01 ppm and <1.00 ppm, report 2 significant figures.

16.2.3.3 For sample concentrations ≥ 1.00 ppm, report 3 significant figures.

16.2.3.4 For QC always report one additional significant figure.

17.0 DATA REPORTING AND DELIVERABLES

17.1 See appendices F, H, I, and J for data reporting.

18.0 QUALITY ASSURANCE

18.1 All quality control data should be maintained and available for easy reference or inspection.

18.2 Linear range studies must be performed every year or when there is a significant change in the instrument response.

18.2.1 To perform a linear range study, calibrate the instrument as it would be calibrated for an actual analysis.

18.2.2 Run standards at high concentrations and calculate the percent recovery for each element. A good place to start is 1 ppm.

18.2.3 If the percent recovery is acceptable (90%-110%), run a standard at a higher concentration. If the percent recovery is not acceptable, run a standard at a lower concentration.

18.2.4 The last standard concentration that had a percent recovery within the acceptance window will be designated as the linear range for that element.

18.3 IDL/MDL studies must be performed on an annual basis.

18.3.1 Instrument Detection Limits (IDLs) in ug/L can be estimated by calculating the average of the standard deviation of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples).

18.3.2 To calculate an MDL, aspirate ten vials of a low concentration standard, typically at a concentration at or near the detection limit. Calculate the standard deviation of a small population and multiply the result by the Student's T value for (n-1) degrees of freedom. If the concentration of the standard ran is greater than ten times the calculated MDL, and the concentration of the standard ran is above the detection limit reported to clients, the MDL must be performed at lower concentration. If the standard concentration is less than ten times the calculated MDL or if the standard used for the analysis was at or below the lowest reporting limit on LIMS, the MDL is acceptable. There is also a spreadsheet located on the computer network that will perform this calculation for you. Please see the metals lab manager if you need assistance in using this program.

18.4 Dilute and reanalyze samples that are more concentrated than the established linear range.

18.5 Include a minimum of one laboratory blank per sample batch to determine if contamination or any memory effects are occurring. This laboratory blank must be carried through the sample preparation procedure.

18.6 Analyze one matrix spike (MS) and one matrix spike duplicate (MSD) at a frequency of at least 1 in 20 (5%). MS and MSDs are aliquots of sample into which a known quantity of analyte is pipetted in. The percent recovery of the analyte is calculated. This measures the accuracy of the sample preparation method as well as the effect of the matrix on the analysis. The relative percent difference is calculated from the MS and MSD concentrations. The MSD checks the precision of the method. The percent recoveries should be between 70 percent and 130 percent of the spiked value.

18.6.1 Pretreated samples must be spiked at the time of digestion before the digestion has begun. Please see the specific pretreatment procedure for a discussion on spiking procedures.

18.6.2 Samples that require dilutions that have an MS/MSD performed on them must also have an MS/MSD performed on an aliquot of dilution.

18.6.3 Samples not requiring pretreatment are spiked before they are physically loaded on the tray.

18.6.3.1 See Appendix A for the method used to spike samples.

18.6.3.2 If the method in Appendix A is followed when spiking you should get 40 ug/L as a final concentration.

18.6.4 Calculate the percent recovery of the MS and MSD as follows,

$$\%R = \frac{C_{Spike} - C_{Orig}}{\text{Spike Quantity}} * 100$$

where:

%R = percent recovery of the spike
C_{Spike} = concentration of the spiked sample
C_{Orig} = original concentration of the sample (the unspiked sample concentration)
Spike Quantity = quantity of the element spiked into the sample

18.6.5 Calculate the relative percent difference between the MS and MSD as follows:

$$\%RPD = \frac{C_{MSD} - C_{MS}}{(C_{MSD} + C_{MS})/2} * 100$$

where:

%RPD = relative percent difference between the MS and MSD
C_{MSD} = concentration of the MSD as read from the raw data printout
C_{MS} = concentration of the MS as read from the raw data printout

18.7 It is recommended that whenever a new or unusual sample matrix is encountered, a series of tests be performed prior to reporting concentration data for analyte elements. These tests, as outlined in 18.7.1 through 18.7.3, will ensure the analyst that neither positive nor negative interferences are operating on any of the analytes to distort the accuracy of the reported values.

18.7.1 **Serial dilution**: If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrument detection limit after dilution), an analysis of 5X dilution must agree within 10% of the original determination. If not, a chemical physical interference effect should be suspected. And standard addition must be performed.

18.7.2 **Matrix spike addition**: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered within 75% to 125% of the known value. The spike addition should produce a minor level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specific limit, matrix effect should be suspected. The use of a standard-addition analysis procedure can usually compensate for this effect.

18.7.3 **Standard addition**: The standard-addition technique involves adding known amounts of standard to one or more aliquots of the process sample solution. This technique compensates for a sample constitute that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct additive interferences which

could a baseline shift. The simple version of this technique is the single addition method, in which identical aliquots of the sample solution, each of volume V_X, are taken. To the first (labeled A) is added a small volume V_S of a standard solution of concentration C_S. To the second (labeled B) is added the same volume V_S of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration C_X is calculated:

$$C_X = \frac{S_B * V_S * C_S}{(S_A - S_B) * V_X}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_S and C_S should be chosen so that S_A is roughly twice S_B on the average. It is best if V_S is made much less than V_X, and thus C_S is must greater than C_X, to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure. For the results of this technique to be valid, the following limitations must be taken into consideration;

1. The analytical curve must be linear.
2. The chemical form of the analyte added must respond the same way as the analyte in the sample.
3. The interference effect must be constant over the working range of concern.
4. the signal must be corrected for any additive interference.

The intensity of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate.

NOTE: A method of standard addition must be performed on any TCLP sample whose spike recovery is not greater than 50%.

18.8 Check the instrument standardization by analyzing appropriate quality control check standards as follows.

NOTE: Steps 18.8.2 through 18.8.6 must be performed every time the instrument is calibrated.

18.8.1 Calibrate the instrument using a standard and a blank.

18.8.2 An ICS/ICV (Initial Calibration Standard/Initial Calibration Verification) is placed after the calibration standards and blank. Percent recovery limits are 90% to 110% of the original calibration standard concentration. If the percent recovery for this standard is not within the acceptance limits for an element that must be analyzed, terminate the analysis, correct the problem, recalibrate, and restart the analysis.

18.8.3 An ICB/CCB (Initial Calibration Blank/continuing Calibration Blank) is analyzed after the ICV. The results of this blank are to be within ± the detection limit. If the blank is not within

± the detection limit for an element that must be analyzed, terminate the analysis, correct the problem, and restart the analysis from the beginning.

18.8.4 The interference check solutions is analyzed at the beginning and end of each run and every 12 hours of instrument operation. This standard is analyzed to demonstrate the magnitude of interferences and provide an adequate test of any corrections. Percent recovery limits are 80% to 120% of the actual concentration. If the percent recovery for this standard is not within acceptance limits. Stop the run remake the standard and reanalyze. If it is now within limits continue the analysis stop the run remake the standard and reanalyze. If it is now within limits continue the analysis. If the standard is still outside of the acceptance limits stop the run, correct the problem, and restart the analysis from the beginning.

18.8.5 An LCS (Laboratory Check Standard), which must come from a different primary source than the calibration standards, is placed after the first CCV in each run. Percent recovery limits are 80% to 120% of the concentration unless control limits have been established. If the percent recovery for this standard is not within the acceptance limits for an element that must be analyzed, terminate the analysis, correct the problem, recalibrate, and restart the analysis from the beginning. See the following table for a tabulation of the LCS concentrations as of this writing. Dashes in the table indicate that the element to the left is not present in the solution.

LCS Concentration When Diluted According to Instructions

Element	SP1036 (ug/L)	SP1037 (ug/L)	ERA 9965
Aluminum	80	---	59.3
Antimony	---	80	11.1
Arsenic	80	---	5.19
Barium	80	---	29.6
Beryllium	80	---	8.89
Cadmium	80	---	8.15
Chromium	80	---	23.0
Cobalt	80	---	55.6
Copper	80	---	44.8
Lead	80	---	43.0
Manganese	80	---	16.3
Molybdenum	---	80	20.7
Nickel	80	---	31.9
Selenium	80	---	17.8
Silver	80	---	8.89
Thallium	80	---	5.19
Vanadium	80	---	11.7
Zinc	80	---	47.4

18.8.6 Verify the calibration after every 10 samples.

18.8.6.1 A CCV (Continuing Calibration Verification) is analyzed after every 10 samples, or sooner. Percent recovery limits are 90% to 1110% of the concentration. If the

percent recovery for this standard is not within the acceptance limits for an element that must be analyzed, terminate the analysis, correct the problem, recalibrate, and restart the analysis from the last good CCV or ICS/ICV.

18.8.6.2 A CCB (Continuing Calibration Blank) is analyzed after the CCV. The results of this blank are to be within ± the detection limit. If the blank is not within ± the detection limit for an element that must be analyzed, terminate the analysis, correct the problem, recalibrate, and restart the analysis from the last good CCB or ICB/CCB.

18.8.7 At the end of each run, verify the calibration.

18.8.7.1 A CCV (Continuing Calibration Verification) is analyzed at the end of each run. Percent recovery limits are 90% to 110% of the concentration. If the percent recovery for this standard is not within the acceptance limits for an element that must be analyzed, terminate the analysis, correct the problem, recalibrate, and restart the analysis from the last good CCV or ICS/ICV.

18.8.7.2 A CCB (Continuing Calibration Blank) is analyzed after the CCV. The results of this blank are to be within ± the detection limit. If the blank is not within ± the detection limit for an element that must be analyzed, terminate the analysis, correct the problem, recalibrate, and restart the analysis from the last good CCB or ICB/CCB.

18.8.7.3 An interference check solution is analyzed at the end of the run. This standard is analyzed to demonstrate the magnitude of interferences and to provide an adequate test of any collections. Percent recovery limits are 80% to 120%. If the percent recovery is not within acceptance limits. Stop the run, remake the standard, and reanalyze. If it is now within limits continue the analysis. If it is still outside of the acceptance limits, stop the run, correct the problem, and restart the analysis from the beginning.

18.9 Internal standardization must be used in all analysis to correct for instrument drift and physical interferences. A minimum of five internal standards are used at all times. For any sample that is to be run by method 200.8, the recovery requirement for each internal standard is 60-125% of the true value. For any sample analyzed by method 6020, the recovery limits are 30-120%.

18.9.1 If the internal standard recovery is low, the sample must be diluted 1:5 and reanalyzed. If the internal standard recovery is still low, a further 1:5 dilution is required. This process is repeated until satisfactory recoveries are achieved.

18.9.2 If the internal standard recovery is high, then the internal standard compound may naturally be present in the sample, and a different internal standard must be used.

19.0 ANALYST CERTIFICATION/METHOD VALIDATION

19.1 Please see section 3.8.3 and 3.9.3.2 of the TriMatrix Quality Assurance Manual and Appendix E for analyst and method certification/validation protocols.

20.0 REFERENCES

20.1 ELAN 6000 Software Guide; Perkin Elmer Corporation; Manual 0993-8965, Release E, June 1995.

20.2 ELAN 6000 Hardware Guide; Perkin Elmer Corporation; Manual 0993-8969, Release D; September 1995.

21.0 ATTACHMENTS/APPENDICES

21.0 See Attached

Approved By: RL 4/23/98 Approved By: 11/23/98
QA Manager Area Manager

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Mercury Atomic Absorption
For the Analysis of Solid or Semisolid Waste
Automated Cold-Vapor Technique

METHOD 7471A

APPROVALS:

Metals Supervisor: Betty C Doyle Date: 1/30/96

QA/QC Supervisor: [signature] Date: 1-30-96

Laboratory Manager: [signature] Date: 1-30-96

Procedure Number: GR-01-109

Revision Number: 2.0 By: Betty Doyle Effective Date: 1/30/96

Total Number of Pages: 41 Pages Revised: All

Subject:	Mercury Atomic Absorption For the Analysis of Solid or Semisolid Waste Automated Cold-Vapor Technique	Procedure No: GR-01-109 Revision No: 2.0 Effective Date: 1/30/96
	USEPA Method 7471	Page 1 of 41

1.0 PRINCIPAL METHOD REFERENCE

1.1 U.S. Environmental Protection Agency. Mercury in Solid or Semi-solid Waste (Manual Cold Vapor Technique) Method 7471A, SW-846 Test Methods for Evaluating Solid Waste, 3rd Edition, Revision 1, September 1994

2.0 PARAMETER LIST

Hg

3.0 SCOPE AND APPLICATION

Method 7471 is used for the determination of total mercury (inorganic and organic) in soil, sludge, oil, hazardous waste. All samples analyzed by this method must undergo a dissolution procedure to destroy any organic substances in the sample.

4.0 SUMMARY OF TEST METHOD

Prior to analysis, all samples, standards, and quality control samples must be prepared according to the procedure discussed in Section 12 of this method. This digestion procedure breaks down organo-mercury compounds into inorganic mercury. The inorganic mercury is then converted to the Hg^{+2} state for subsequent analysis.

The prepared liquid sample with mercury in the divalent form (Hg^{+2}) enters the mercury analysis system and is mixed with stannous sulfate to form elemental mercury vapor (Hg^0) according to the following equation:

$$Hg^{+2} + Sn^{+2} \text{-------} > Hg^0 + Sn^{+4}$$

The mixture flows into a liquid-gas separator where argon is introduced to carry the mercury through a drying tube containing magnesium perchlorate. The dry mercury vapor then enters one path of a heated double-path optical cell which has been optimized for fast response time. A mercury source powered by a constant current power supply delivers a stable source of emission at 253.7 nm. Absorbance by the mercury vapor is measured

using a solid state detector with a wide dynamic range. The resulting signal is referenced to the simultaneous absorbance of the pure carrier gas flowing through the second optical path under identical conditions. The absorbances of the standards are plotted against the known concentrations. Absorbance readings from unknown samples are then read from the standard curve and extrapolated to the concentration. The mercury vapor is then passed through activated carbon prior to being vented up a hood to reduce the mercury emissions from this test.

5.0 INTERFERENCES AND CORRECTIVE PROCEDURES

5.1 Soil samples often contain mercury at low levels. The preparation area must be kept free of dust and dirt to avoid contamination.

5.2 Certain volatile organic materials that absorb at this wavelength (253.7 nm) may also cause interference. If an interference is suspected, a preliminary run without reagents should determine if this type of interference is present.

5.3 The inorganic lab uses a product called Chrom-merge which contains mercury at fairly high concentrations to clean BOD bottles. Since this method uses the same type of bottles as the BOD test, the analyst should ensure that the bottles used for this test are 'Metals' bottles and that they have not been washed with the reagent. All BOD bottles used for mercury have an 'M' etched on the side of the bottle. If the bottles have been contaminated with the reagent, all suspected bottles must be thoroughly washed before use.

5.4 If a thermometer is broken anywhere in the laboratory, the metals lab will be notified. The possibility exists that sample bottles may become contaminated from the mercury vapor from the broken thermometer. If contamination is suspected, the contents of the bottles must be disposed of and the samples redigested.

5.5 Mercury may be lost from samples during drying if the temperature of the oven exceeds 60°C. All solid samples must be dried at 60°C and homogenized prior to digestion.

5.6 Mercury may be lost if the temperature of the water bath used to perform the digestion goes above 95°C. All water baths used for digestion must have their temperature

monitored with an alcohol thermometer. The temperature of the water bath must be recorded in the mercury digestion logbook.

5.7 The reagents used in this procedure may become contaminated over time. If contamination is suspected, the reagent must be discarded and fresh reagent made.

6.0 SAFETY PRECAUTIONS

6.1 The analyst must comply with all standard operating procedures for health and safety as outlined in the "TriMatrix Laboratory Safety Manual".

6.2 Concentrated acids are used in the preparation of standards and samples for analysis by cold vapor. Gloves and safety glasses must be worn at all times when handling concentrated acids. Gloves must also be worn when handling digested samples. Please refer to the MSDSs for information on these or any other chemicals utilized in this procedure.

6.3 Check the exhaust hood over the instrument to be sure it is operating correctly. If the ventilation system is not working properly, immediately contact the metals lab supervisor. Under no circumstances should the cold vapor unit be used if the exhaust hood is not working.

6.4 UV protective glasses must be worn at all times in the metals laboratory. The mercury lamp emits UV radiation. Avoid looking directly at the lamp without some type of strong UV protection. Failure to follow this policy may cause very serious and immediate damage to the retina of the eye.

6.5 The cold vapor unit uses Argon (Ar) as the carrier gas for the mercury vapor. Although Ar in and of itself is neither hazardous nor flammable, it may cause suffocation through oxygen deprivation. It is therefore imperative that all sources of Ar be turned off with a valve when not in use. Since Ar is colorless and odorless, if you feel lightheaded, please evacuate the metals lab at once and notify the metals lab supervisor. Please refer to the MSDSs for information on this or any other chemicals utilized in this procedure.

6.6 Mercury may exist in many forms, and is toxic in a variety of ways. Mercury vapor is toxic if inhaled. Do not operate the instrument if it is not properly ventilated. Hg^{+2} is toxic if ingested. Always wash hands after handling the mercury standard.

6.7 Many of the reagents used in this procedure are toxic if ingested. Please refer to the MSDSs for information on the chemicals utilized in this procedure.

6.8 No food or drink is allowed in the metals lab. Food or drink may become contaminated with acid or metals and may therefore be hazardous.

6.9 Wash hands before starting work. Chemicals may be present on the skin which may interfere with metals analysis. Wash hands before leaving the metals lab. Chemicals and acids may be on the skin which could eventually be ingested or passed on to a third party through casual contact.

7.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

7.1 All sample containers must be pre-washed with detergents, acids and ASTM Type II water. Plastic and glass containers are both suitable, although glass is preferable since mercury may be adsorbed by certain plastics.

7.2 No preservation is required for solid or semi-solid samples. All samples should be refrigerated until there is time to dry the samples and prepare them for analysis.

7.3 For solids or semi-solids, moisture must be driven off in a drying oven at a temperature of $60^{o}C$.

7.4 All samples must be subjected to a dissolution step prior to analysis. For specific steps see Section 10 of this method.

8.0 APPARATUS/INSTRUMENTATION

8.1 300 ml BOD bottles. The bottles must be in good condition and have a ground-glass stopper. If the bottles are weak in any way, they will crack when subjected to the preparation procedure.

8.2 Laboratory analytical balance capable of weighing 0.1 mg.

8.3 Laboratory balance capable of weighing 0.1 g.

8.4 Plastic weigh boats. The weigh boats used must be free of mercury. Usually, the weigh boats are used to weigh the samples and dispense the sample into the BOD bottle. In the case of oil samples and some wastes, the weigh boat is actually placed in the BOD bottle with the sample to reduce weighing errors.

8.5 Eppendorf pipettors capable of delivering 50-1000 µl.

8.6 Class A volumetric flasks, various volumes.

8.7 Class A reusable pipettes, various volumes.

8.8 Oxford pipettes capable of dispensing 5 to 10 mls of solution.

8.9 Hotplates capable of heating water to 95°C and maintaining the temperature of the water at 95°C.

8.10 Roasting pans with lids. The pans must be capable of holding water. The lids should fit securely over the pan, but should not be air-tight.

8.11 Magnetic stir plate and stirbar.

8.12 Laboratory spatulas. The spatulas must be cleaned prior to use.

8.13 Timer capable of counting down from 30 minutes.

8.14 Glass alcohol thermometer. Mercury thermometers must never be used in this procedure. The thermometer must be readable at 95°C.

8.15 Leeman Labs PS200 automated mercury system. The instrument has an attached autosampler that is capable of aspirating sample and reagents. A regulator is built into the instrument to deliver the Ar to the sample stream. The instrument is a double-beam instrument capable of measuring the change in intensity of a mercury lamp at 253.7 nm. The Ar used to purge the mercury vapor from the sample is used as the reference beam. To compensate for any aberrations in the optical path, the instrument has an adjustment screw to tune the path so that they react the same to the emission of the mercury lamp.

8.16 Computer running Leeman PS 200 control software. An IBM compatible 80386SX16 or better computer with at least 1 MB RAM, a 40 MB hard disk, and a VGA color monitor is needed. The current software used by the instrument is PS200 version 2.008. The software version may be updated without notice if it performs at least as well as the older version. Major updates to the software that require the analyst to deviate from the stated procedure will require an update to this SOP.

8.17 Epson printer or equivalent. The specific printer used must respond to Epson printing codes. The printer must be attached to the computer using an appropriate communication cable. Please refer to the Instrument manual for further printer specifications.

9.0 ROUTINE PREVENTIVE MAINTENANCE

9.1 Check calibration on pipettors prior to use. Please refer to the Eppendorf pipettor SOP for calibration procedures and corrective actions.

9.2 Verify calibration of the balance. Please refer to the balance SOP for calibration procedures and corrective actions.

9.3 Inspect the Ar supply when the shift begins. If the liquid level of the Ar falls below the reorder mark, notify the person responsible for ordering gasses or the metals supervisor.

Approved By: SJ 1-30-96 QA/QC Supervisor

Approved By: BJO 1-30-96 Area Supervisor

9.4 Inspect the waste container on the floor under the instrument every shift. If the jug is full, properly dispose of the liquid.

9.5 Inspect the printing quality of the printer. If the print is hard to read or light in appearance, replace the printer cartridge with the appropriate replacement.

9.6 Check all tubing for flattened areas, leaks, and clogs before use. If any problems are found, change the tubing and note the change in the instrument maintenance logbook.

9.7 Prior to analysis, rinse the entire system with 1:3 HCl for about ½ hour followed by ASTM Type II H_2O for another ½ hour.

9.8 The aperture must be checked before the analysis has begun. See the analytical method for a discussion on how the aperture is tested.

9.9 The instrument must be given the appropriate amount of time to warm up before use. If the instrument has been shut down in 'Overnite' mode, it is highly recommended that the 'WARMSTRT' macro be invoked to warm the instrument up. If the instrument has been shut down for an extended period of time, the 'COLDSTRT' macro should be used.

9.9.1 The WARMSTRT macro asks a series of questions to the user to ensure that everything is in place and is in working order. The instrument then warms up for 20 minutes to condition the tubing and stabilize the lamp. When answering the questions, if the user enters an invalid response, the macro will not continue. For example, one of the questions is 'Is the lamp on? <Y/N>'. If the user enters anything but 'Y', the macro will not continue. When the macro has completed the warm-up period, the message 'System Ready' is displayed on the screen. If the macro was called from another macro, such as the MERCURY macro, then control is given back to the calling macro and the calling macro will resume at the step after the call to the macro.

9.9.2 The COLDSTRT macro is similar to the WARMSTRT macro, but the warm-up period is much longer since it will be invoked only if the instrument has been shut

down for an extended period of time. Again, the questions are programmed such that only certain responses will allow the macro to continue. The warm up time for this macro is 2½ hours. When the macro has completed the warm-up period, the message 'Operation Complete' is displayed on the screen. If the macro was called from another macro, such as the MERCURY macro, then control is given back to the calling macro and the calling macro will resume at the step after the call to the macro.

10.0 CHEMICALS AND REAGENTS

10.1 Acids used in the preparation of standards and for sample processing must be reagent grade or better. Redistilled acids may be used if it has been demonstrated that the acid is free from contamination.

10.1.1 Concentrated hydrochloric acid, trace metal grade.

10.1.2 Concentrated nitric acid, trace metal grade.

10.1.3 Concentrated sulfuric acid, trace metals grade.

10.2 **ASTM Type II water** (ASTM D1193). Deionized water is fed into an all glass distillation unit. The resulting distillate is immediately placed into a plastic container. Impurities are measured by the Inorganic group at TriMatrix. This is the only water acceptable for use in the Metals lab for dilutions or standard preparation.

10.3 **Standard stock solutions** are purchased primarily from Inorganic Ventures. All stock solutions are ICP grade single-element solutions at concentrations of 1000 or 10000 ppm.

10.4 Argon gas supply: Welding grade or better. Currently, this is plumbed from a liquid Ar tank located outside of the building. Ar is used as the carrier gas for analysis.

10.5 Aqua Regia: Prepare immediately before use in a hood by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3. This solution should be

constantly stirred during use on low speed to reduce the amount of gas that the pipettor picks up. **CAUTION:** Chlorine gas is constantly liberated from this solution. The solution may only be used under a hood. If chlorine is detected outside of the hood, try to correct the problem by checking all sample bottles.

10.6 **Sulfuric Acid 0.5N.** Rinse a clean 1 liter volumetric flask several times with ASTM Type II water. Place about 500 ml ASTM Type II water in the flask. Carefully add 14 ml concentrated sulfuric acid to the water. Carefully swirl the flask to mix the solution. Dilute to the mark with ASTM Type II water. Note: the addition of H_2SO_4 to water will cause a considerable amount of heat to be generated. Mix well.

10.7 Potassium Permanganate, 5% solution (W/V). In a plastic 3-liter container, place 150 g $KMnO_4$ crystals. Add 3 L ASTM Type II water to the container. Place a stirbar in the solution and stir on a stirplate until all of the crystals have dissolved. Since this solution is nearly a saturated solution, it may take several hours to prepare. To check if the crystals have dissolved, a glass pipette may be used to probe the bottom of the container for excess crystals. If any of the solution is spilled, immediately clean up the spill with water. If a brownish stain remains, it may be removed from surfaces with hydroxylamine hydrochloride. If the solution touches exposed skin, the skin will turn brown. The brown discoloration is not harmful as long as the excess permanganate is washed off. The stain will wear off over time. Potassium permanganate will collect mercury if left exposed to the atmosphere. The container must be covered when not in use and should be stored in a dark cabinet to reduce the potential for auto-catalytic reduction.

10.8 Sodium Chloride Hydroxylamine Hydrochloride, 12% solution (W/V). In a plastic 3-liter container, place 360 g of sodium chloride and 360 g of hydroxylamine hydrochloride. Add 3 L of ASTM Type II water and a stirbar. Place the container on a stirplate and stir until all of the crystals have dissolved.

10.9 Stannous Sulfate, 10% solution (W/V). Rinse a clean 500 ml volumetric flask several times with ASTM Type II water. Place about 250 ml 0.5 N H_2SO_4 in the flask. Add 50 g stannous sulfate and dilute the flask to volume with 0.5 N H_2SO_4. Mix well. When the crystals have gone into solution, filter through a 0.45 µm filter by using a vacuum apparatus and a side arm filtering flask. **NOTE:** This mixture is a suspension and must be stirred continuously during use, even after filtration.

11.0 STANDARDS

11.1 All standards are single-element standards. All standards are good for 1 day.

11.2 Preparation of the 10,000 ppb standard.

11.2.1 Rinse a clean 100 ml volumetric flask with ASTM Type II water several times.

11.2.2 Place about 20 ml of ASTM Type II water in the flask.

11.2.3 Add 0.5 ml concentrated trace metal grade HNO_3 to the flask and swirl.

11.2.4 Pipette 1 ml 1000 ppm stock standard solution into the volumetric flask.

11.2.5 Dilute the flask to the mark and mix well.

11.2.6 Update the stock standard logbook.

11.2.7 Transfer the standard number from the standard logbook to the volumetric flask.

11.3 Preparation of the 1000 ppb standard.

11.3.1 Rinse a clean 100 ml volumetric flask with ASTM Type II water several times.

11.3.2 Place about 20 ml of ASTM Type II water in the flask.

11.3.3 Add 0.5 ml concentrated trace metal grade HNO_3 to the flask and swirl.

11.3.4 Pipette 10 ml 10,000 ppb stock standard solution prepared in Step 11.2 into the volumetric flask.

11.3.5 Dilute the flask to the mark and mix well.

11.3.6 Update the stock standard logbook.

11.3.7 Transfer the standard number from the standard logbook to the volumetric flask.

11.4 Preparation of the 100 ppb standard.

11.4.1 Rinse a clean 100 ml volumetric flask with ASTM Type II water several times.

11.4.2 Place about 20 ml of ASTM Type II water in the flask.

11.4.3 Add 0.5 ml concentrated trace metal grade HNO_3 to the flask and swirl.

11.4.4 Pipette 1 ml 10,000 ppb stock standard solution prepared in Step 11.2 into the volumetric flask.

11.4.5 Dilute the flask to the mark and mix well.

11.4.6 Update the stock standard logbook.

11.4.7 Transfer the standard number from the standard logbook to the volumetric flask.

11.5 Preparation of working standards.

11.5.1 Set aside 12 clean BOD bottles for the mercury working curve.

11.5.2 Label the BOD bottles as follows:

5.0 ppb ---------- 2 bottles
2.0 ppb ---------- 2 bottles
1.0 ppb ---------- 2 bottles
0.5 ppb ---------- 2 bottles
0.2 ppb ---------- 2 bottles
Blank ---------- 2 bottles

11.5.3 Pipet 5 ml of the 100 µg/L Hg standard into each of the BOD bottles labeled 5.0 ppb. Add 5 ml ASTM Type II water to give a final volume of 10 ml in each BOD bottle.

11.5.4 Pipet 2 ml of the 100 µg/L Hg standard into each of the BOD bottles labeled 2.0 ppb. Add 8 ml ASTM Type II water to give a final volume of 10 ml in each BOD bottle.

11.5.5 Pipet 1 ml of the 100 µg/L Hg standard into each of the BOD bottles labeled 1.0 ppb. Add 9 ml ASTM Type II water to give a final volume of 10 ml in each BOD bottle.

11.5.6 Using a calibrated Eppendorf pipette, pipet 0.5 ml of the 100 µg/L Hg standard into each of the BOD bottles labeled 0.5 ppb. Add 9.5 ml ASTM Type II water to give a final volume of 10 ml in each BOD bottle.

11.5.7 Using a calibrated Eppendorf pipette, pipet 0.1 ml of the 100 µg/L Hg standard into each of the BOD bottles labeled 0.1 ppb. Add 9.9 ml ASTM Type II water to give a final volume of 10 ml in each BOD bottle.

11.5.8 Add 10 ml ASTM Type II water to each of the BOD bottles labeled Blank.

11.5.9 All working standards must be digested within 8 hours of preparation.

11.5.10 Complete the standards log book. See Appendix J.

11.6 Preparation of the Laboratory Fortified Blank (LFB).

11.6.1 Label 2 clean BOD bottles as LFB.

11.6.2 Add 10 ml ASTM Type II to each of the bottles labeled LFB.

11.6.3 Using a calibrated Eppendorf pipette, pipet 0.25 ml of the 1000 µg/L Hg standard into each of the BOD bottles labeled LFB.

11.7 Preparation of the Laboratory Control Sample (LCS).

11.7.1 Label 2 clean BOD bottles as LCS.

11.7.2 Add 10 ml ASTM Type II to each of the bottles labeled LCS.

11.7.3 Pipette 1 ml of the 3 ppm concentrated LCS sample (SP1035) obtained from SPEX into each of the bottles labeled LCS. Note: a concentrated LCS from another vendor or of another concentration may be substituted for the above LCS, as long as the LCS is from a different source than the primary standards and the final concentration of the diluted LCS is around the midpoint of the calibration curve.

12.0 SAMPLE PREPARATION

12.1 Solid sample Preparation

12.1.1 Weigh triplicate 0.2g portions of untreated sample ~~All samples must be prepared in triplicate. Matrix spikes and matrix spike duplicates must also be prepared in triplicate.~~ BOD bottles

12.1.2 All soils, sludges, and sediments must be dried and homogenized prior to digestion by this method. See Appendix M for a discussion on drying and crushing soils.

12.1.3 Wastes, liquid wastes, oils, solvents, TCLP oil phases, and other non-soil sample types are analyzed as-is after homogenization. Weigh triplicate 0.1g portions BOD bo

12.1.4 Generate a worklist for mercury digestion from LIMS. See Appendix F.

12.1.5 Label 1 BOD bottles with the sample number. If the sample will have matrix QC performed on it, then label 2 additional BOD bottles, 1 as the sample SPK, and 1 as the sample MSD. Make sure that the sample number is located somewhere on SPK and MSD bottles.

12.1.6 Weigh out the sample

triplicate 0.1g portions

12.1.6.1 For waste or sludge samples, weigh ~~0.100 g~~ of sample in a plastic weigh boat. Transfer the sample to the BOD bottle. If the sample has obviously adhered to the weigh boat, push the weigh boat into the bottle with the sample. Label the bottle with the sample number and the dilution factor ~~(X1000)~~

triplicate 0.2g portions

12.1.6.2 For soil or soil-like samples, weigh ~~0.200 g~~ of sample in a plastic weigh boat. Transfer the sample to the BOD bottle. If the sample has obviously adhered to the weigh boat, push the weigh boat into the bottle with the sample. Label the bottle with the sample number and the dilution factor ~~(X500)~~

12.1.7 Repeat Step 12.1.6 for the remaining bottles for the sample. Discard the weigh boat when the sample is finished.

12.1.8 Repeat Steps 12.1.5 and 12.1.6 for the remaining samples in the digestion batch.

12.1.9 Add 15 ml ASTM Type II water to each sample bottle.

12.1.10 In a fume hood, add 5 ml aqua regia to each bottle.

12.1.11 Plunge the bottles into a water bath that is maintained at 95°C. Cook the samples for 2 minutes. The temperature of the water bath must be monitored using a thermometer. Note the temperature of the water bath in the mercury digestion logbook.

NOTE: If any of the bottles have been weakened, the bottoms may fall off the bottle. If the bottle breaks, immediately prepare another bottle of the sample.

12.1.12 Cool the samples on the counter until cool or warm to the touch.

12.1.13 Add 50 ml ASTM Type II water to each bottle. Mix well by swirling the bottle after the addition.

12.1.14 Add 15 ml of the 5% potassium permanganate solution to each bottle. Mix well by swirling the bottle after the addition.

12.1.15 Plunge the bottles into a water bath that is maintained at 95°C. Cook the samples for 30 minutes. The temperature of the water bath must be monitored using a thermometer. Note the temperature of the water bath in the mercury digestion logbook

NOTE: If any of the bottles have been weakened, the bottoms may fall off the bottle. If the bottle breaks, immediately prepare another bottle of the sample.

12.1.16 Cool the samples on the counter until cool or warm to the touch.

12.1.17 In a fume hood, add 6 ml of the 12% hydroxylamine hydrochloride solution to each bottle. Mix well by swirling the bottle after the addition.

NOTE: The addition in 12.1.17 will generate large quantities of N_2 gas. Since there is Cl_2 gas in the headspace of the bottle, the liberation of N_2 will push the Cl_2 out of the bottle. Therefore, this addition must be done in a hood. For some samples, the effervescence may become quite vigorous and the stopper may be blown off the BOD bottle.

12.1.18 While the samples are still in the fume hood, add 50 ml ASTM Type II water to each bottle. Mix well by swirling the bottle after the addition.

12.1.19 Arrange the bottles on the counter so that the three bottles for each sample are next to each other.

12.1.20 Print out a LIMS pretreatment benchsheet. See Appendix F.

12.1.21 Pretreat the samples on LIMS. See Appendix C.

12.1.22 Fill out the mercury digestion logbook. See Appendix D.

12.2 Working standard Preparation

12.2.1 Prepare the working standards as described in Section 11.5.

12.2.2 Prepare an LCS for the analysis by pipetting an appropriate amount of a concentrated LCS solution into a BOD bottle so that the final concentration of the LCS is about equal to the midpoint of the calibration curve. When calculating the amount of spike needed, use 100 ml for the final volume.

12.2.3 In a fume hood, add 5 ml aqua regia to each bottle of standard and LCS.

12.2.4 Plunge the bottles into a water bath that is maintained at 95°C. Cook the standards for 2 minutes. The temperature of the water bath must be monitored using a thermometer. Note the temperature of the water bath in the mercury digestion logbook.

NOTE: If any of the bottles have been weakened, the bottoms may fall off the bottle. If the bottle breaks, immediately prepare another bottle of the standard or LCS.

12.2.5 Cool the standards and LCS on the counter until cool or warm to the touch.

12.2.6 Add 50 ml ASTM Type II water to each bottle. Mix well by swirling the bottle after the addition.

12.2.7 Add 15 ml of the 5% potassium permanganate solution to each bottle. Mix well by swirling the bottle after the addition.

12.2.8 Plunge the bottles into a water bath that is maintained at 95°C. Cook the standards and LCS for 30 minutes. The temperature of the water bath must be monitored using a thermometer. Note the temperature of the water bath in the mercury digestion logbook.

NOTE: If any of the bottles have been weakened, the bottoms may fall off the bottle. If the bottle breaks, immediately prepare another bottle of the sample.

12.2.9 Cool the standards and LCS on the counter until cool or warm to the touch.

12.2.10 In a fume hood, add 6 ml of the 12% hydroxylamine hydrochloride solution to each bottle. Mix well by swirling the bottle after the addition.

NOTE: The addition in 12.2.9 will generate large quantities of N_2 gas. Since there is Cl_2 gas in the headspace of the bottle, the liberation of N_2 will push the Cl_2 out of the bottle. Therefore, this addition must be done in a hood. For some samples, the effervescence may become quite vigorous and the stopper may be blown off the BOD bottle.

12.2.11 While the standards and LCS are still in the fume hood, add 50 ml ASTM Type II water to each bottle. Mix well by swirling the bottle after the addition.

12.2.12 Fill out the mercury digestion logbook. See Appendix D.

13.0 CALIBRATION PROCEDURES (INSTRUMENTAL ANALYSIS)

13.1 Calibration of the instrument must be performed every 24 hours or for every batch of samples that were prepared in the same manner, whichever is more frequent. For example, if a soil calibration was performed at 8 AM and a water run is to be performed at 5 PM of the same day, a new calibration curve must be performed with standards prepared with the samples to be analyzed.

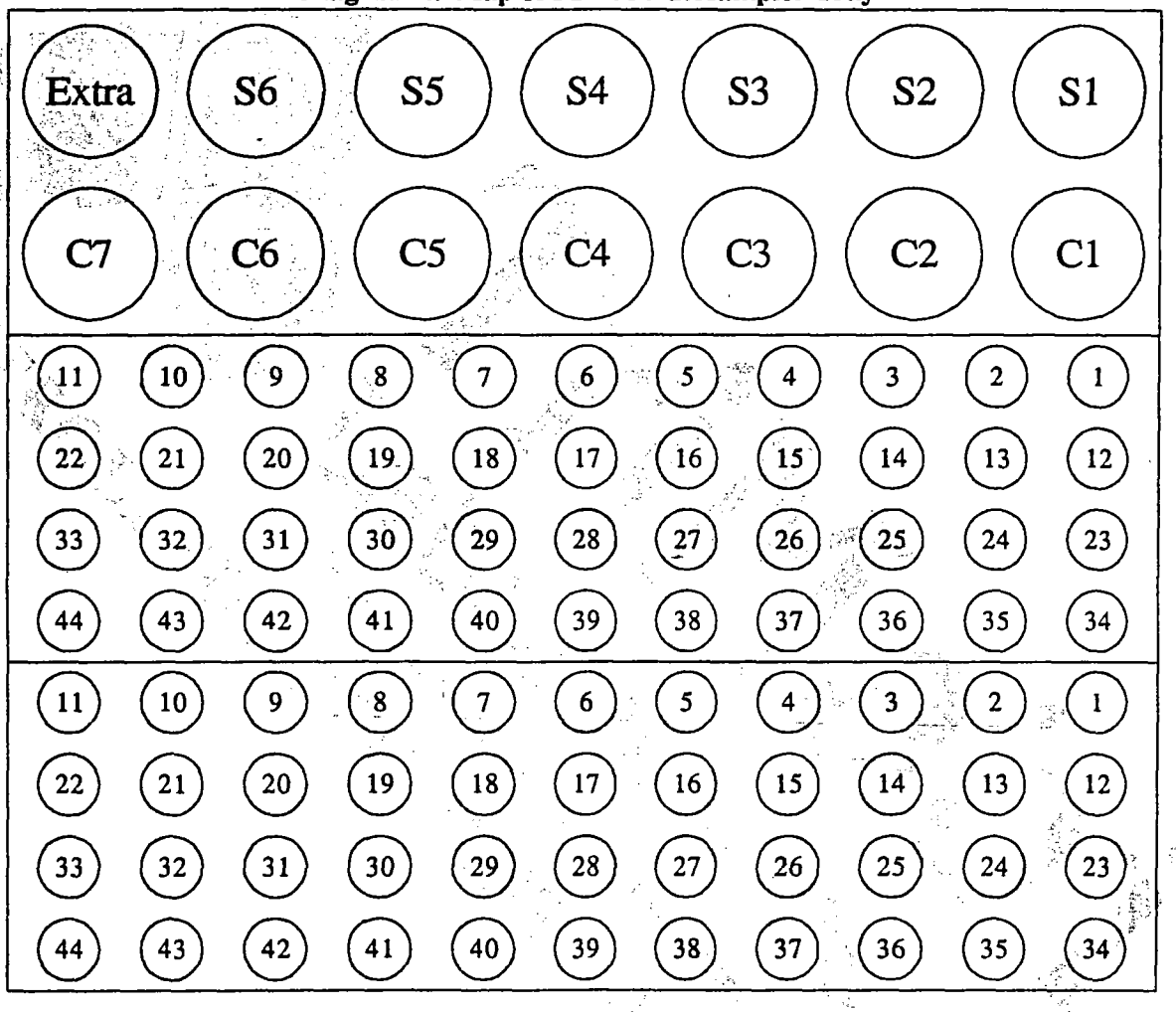
13.2 Start the instrument software as described in Section 14. Perform a COLDSTRT or a WARMSTRT, whichever is appropriate.

13.3 Load the autosampler tray with the appropriate standards. See Table 1 for autosampler positions. Rack 0 is the rack that is furthest away from the front of the instrument. See Diagram 1 for autosampler position designation.

Table 1. Autosampler Positions for Standards

Autosampler Position	Standard Concentration (ppb)
Rack 0, Position 1 (S1)	0 (Blank)
Rack 0, Position 2 (S2)	0.1
Rack 0, Position 3 (S3)	0.5
Rack 0, Position 4 (S4)	1.0
Rack 0, Position 5 (S5)	2.0
Rack 0, Position 6 (S6)	5.0

Diagram 1. Map of PS-200 Autosampler Tray



13.4 There are currently three ways to calibrate the instrument. Although the three methods are different in the keystrokes used, they are essentially performing the same task.

13.4.1 Method 1: Allow the MERCURY macro to run the standards unattended. This method will automatically initialize all parameters, run the standards, check the QC, run samples, and run periodic QC.

13.4.1.1 Start the MERCURY macro. When the PS200 software is booted up, the MERCURY macro is automatically invoked. If, for some reason the macro is stopped, the macro can be restarted by pressing <F2> then typing in MERCURY and pressing <Enter>.

13.4.1.2 Follow the direction outlined in Section 14 until the software asks if standards should be ran. Answer 'Y' to this question.

13.4.1.3 The instrument will automatically run the standards and plot the calibration curve when finished. Skip to Step 13.5.

13.4.2 Method 2: Use the RUNSTDS macro to run the standards unattended. This method will run the standards unattended and then stop. The user will have to either invoke the MERCURY macro again to restart the analysis in unattended mode or else the run will have to be performed manually.

13.4.2.1 Set up the instrument as described in Section 14. Perform a WARMSTRT or a COLDSTRT, if needed.

13.4.2.2 Start the macro by pressing <F2>, then typing in RUNSTDS and pressing <Enter>.

13.4.2.3 The instrument will automatically run the standards and plot the calibration curve when finished. Skip to Step 13.5.

13.4.3 Method 3: Run each standard individually. This method needs constant operator attendance and should not be used if the entire curve is to be ran. The operator is responsible for checking all values before running the analysis if this method is used.

13.4.3.1 Press <F6> (Standard).

13.4.3.2 The software will ask for the standard number to run. Enter the standard number that should be analyzed. Do not enter the standard concentration,

only the standard position. Refer to Diagram 1 for the standard autosampler position.

13.4.3.3 Repeat steps 13.4.3.1 and 13.4.3.2 for the remaining standards that need to be ran.

13.4.3.4 When finished running the standards, go to the calibration page by pressing <F1> (Main Menu) then pressing <C> (Calibration) <L> (Line calibration). At this point a plot of the calibration should be displayed.

13.4.3.5 Press <A> to accept the calibration curve.

13.5 The correlation coefficient 'r' must be equal to or greater than 0.995. If the correlation coefficient is not equal to or greater than 0.995, you may edit the calibration curve.

13.5.1 Rerun a point in the calibration curve.

13.5.1.1 Press <F6> (Standard).

13.5.1.2 The software will ask for the standard number to run. Enter the standard number that should be analyzed. Do not enter the standard concentration, only the standard position. Refer to Diagram 1 for the standard autosampler position.

13.5.1.3 Repeat steps 13.5.1.1 and 13.5.1.2 for the remaining standards that need to be ran. **NOTE:** The software will not automatically re-calculate the correlation coefficient. The user must press <A> to re-calculate the coefficient.

13.5.2 Delete a point in the curve that is not correct. Since it is possible that there was a determinate error in the preparation of the standards, standard points may be dropped if they appear to significantly deviate from the curve. At least 3 standards, one of which is the lowest standard of the

curve, and the blank must remain in the curve for the calibration curve to be valid.

13.5.2.1 Use the arrow keys to highlight the point that is to be deleted from the curve.

13.5.2.2 Press <Enter> to delete the point from the calibration curve.

13.5.2.3 Press <A> to re-calculate the calibration curve and correlation coefficient.

13.5.3 Add a previously deleted point back to the curve. If a calibration point has been deleted by accident, or if it was deleted to check its affect on the remaining curve, the point may be added back without re-running the point. At least 3 standards, one of which is the lowest standard of the curve, and the blank must remain in the curve for the calibration curve to be valid.

13.5.3.1 Use the arrow keys to highlight the point that is to be added back to the curve.

13.5.3.2 Press <Enter> to add the point to the calibration curve.

13.5.3.3 Press <A> to re-calculate the calibration curve and correlation coefficient.

13.6 If the MERCURY macro was used to produce the analytical curve, press <F2> when the curve is acceptable. The software will double check that the correlation coefficient is correct, then print the analytical curve if the correlation is acceptable. The macro will then automatically start analyzing the quality control samples and samples. See Section 14 for a more in depth discussion on performing the analysis.

13.7 If the RUNSTDS macro was used to produce the analytical curve, press <F2> when the curve is acceptable. The software will double check that the correlation coefficient is correct, then print the analytical curve if the correlation is acceptable. The macro will then end. At this point, the quality control samples must be analyzed. If an unattended mode of operation is preferred, start the MERCURY macro by pressing <F2> and then typing in MERCURY<Enter>. Performing the analysis manually is beyond the scope of this procedure. See Section 14 for a more in depth discussion on performing the analysis.

13.8 If the curve was constructed by manually running the standards, double check that the curve is accepted the curve by pressing <A>. Check to ensure that the correlation coefficient is still 0.995 or greater. If the correlation is not at least 0.995, go back to step 13.5 and edit the calibration. If the correlation is okay, print the curve by pressing <F4> (Print Screen). At this point, the quality control samples must be analyzed. If an unattended mode of operation is preferred, start the MERCURY macro by pressing <F2> and then typing in MERCURY<Enter>. Performing the analysis manually is beyond the scope of this procedure. See Section 14 for a more in-depth discussion on performing the analysis.

14.0 ANALYTICAL PROCEDURE

14.1 This procedure assumes that the user has been introduced to the software either by reading the software manual or by experienced metals personnel. Advanced topics, such as how to replace the mercury source lamp are beyond the scope of this method. In this case, the user should reference the instrument manual.

14.2 Make sure that the argon supply is on. There is a valve behind the instrument that supplies the PS200 instrument. Make sure that the valve is open.

14.3 Turn the instrument on if it is not already on. The power button for the instrument is the blue button on the front of the instrument. If the instrument fails to turn on, contact the metals lab supervisor.

14.4 Turn the computer and printer on if they are not turned on. If the computer fails to boot, contact the metals lab supervisor.

14.5 Turn the lamp on. The lamp power button is the green button on the front of the instrument. Allow the lamp to warm up for at least ½ hour before testing the aperture. If the lamp does not seem to be working, contact the metals lab supervisor.

14.6 The computer will automatically start the MERCURY macro. Stop the macro by pressing <F10> (Stop).

14.7 Place the stannous sulfate line (the line connected to the red/red tubing) and the sample line (the line connected to the black Viton tubing) in the wash vessel. Fill the wash vessel with 10% HCl solution.

14.8 Check the peristaltic tubing. If the tubing is blocked or badly worn, replace it with new tubing.

14.9 Clamp the tubing in place. Make sure that the peristaltic tubing is in a straight line over the pump rollers. Lift the red lever on the pump manifold to press the tubing against the rollers.

14.10 Start the pump. From the **Main Menu** , go to the **Instrument** screen by pressing **I**. Press **O** to go to the **Operation** screen. Press **R** then **O** to start the pump.

14.11 Turn on the Argon carrier gas. While in the **Instrument Operation** screen, press **G** then **O** to turn on the carrier gas.

14.12 Check the peristaltic tubing to ensure that the pump is drawing liquid. Lift the sample line out of the rinse solution to see if the pump will pull air. Check the stannous sulfate line the same way. Watch the drain line right after the liquid/gas separator to see if bubbles are being drawn through the tubing. If none of the tubing is drawing, make sure that the pump is turning. If a single piece of tubing is not drawing, try to increase the tension to make the tubing draw by lifting the red lever on the pump manifold higher. If liquid is still not being drawn, try changing the tubing (See Section 9.6). If the pump still does not draw, a clog

should be suspected. Please see the metals lab supervisor for help in finding and correcting the cause of the clog.

14.13 Fill the drying tube with drying agent. Currently, the metals lab is using Magnesium Perchlorate (Anhydrone) as the drying agent for mercury determinations.

14.13.1 Place a small amount of glass wool in one end of the drying tube. There should be just enough glass wool to keep the drying agent from falling out of the tube. Do not put too much glass wool in the end of the tube as this may restrict gas flow through the drying tube.

14.13.2 While wearing gloves, carefully fill the drying tube with fresh magnesium perchlorate. Fill the tube up to the neck of the tube. Gently tap the bottom of the tube on the counter to help the crystals settle into place. Do not over pack the tube as this will restrict the gas flow through the tube. Fill the tube up again within ¾ inch of the end of the tube.

14.13.3 Place a small amount of glass wool in the end of the drying tube. There should be just enough glass wool to keep the drying agent from falling out of the tube. Do not put too much glass wool in the end of the tube as this may restrict gas flow through the drying tube.

14.13.4 Check the O-rings on the drying tube endcaps for wear. Replace these O-rings if there is obvious wear.

14.13.5 Place the drying tube end caps on the end of the drying tube. Push the ends on until they stop. Tighten the nut on the endcaps until snug. Do not over tighten the endcaps.

14.13.6 Place the drying tube in its holder on the instrument.

14.14 If the lamp has been on for at least ½ hour, test the aperture.

14.14.1 Start the APERTEST macro.

14.14.1.1 Press <F2> (Macro).

14.14.1.2 Type in APERTEST, then press <Enter>.

14.14.2 The macro will now start to display some numbers on the screen. These numbers are the readings for the aperture and they represent the difference in intensity between the sample beam of the instrument and the reference beam.

14.14.3 The aperture reading must be 0±100, preferably 0±20. If the reading is not within specifications, adjust the aperture until the value is within range. Adjustment should be done as quickly as possible so that the temperature of the flow cell does not change significantly.

14.14.3.1 Remove the front cover from the instrument to reveal the flow cell. Push the cover up and then pull forward to remove.

14.14.3.2 Remove the Allen wrench from the front panel. The wrench is used to turn the aperture screws.

14.14.3.3 There are two screws on the flow cell at the right-hand end of the flow cell. Always adjust the screw that has been turned into the flow cell the furthest.

14.14.3.4 If the reading is negative, turn the screw clockwise until the reading is zero.

NOTE: In order to obtain an accurate reading, let go of the Allen wrench while taking the reading. Any pressure applied to the flow cell during the reading will change the reading.

14.14.3.5 If the reading is positive, turn the screw counter-clockwise until the reading is zero.

NOTE: In order to obtain an accurate reading, let go of the Allen wrench while taking the reading. Any pressure applied to the flow cell during the reading will change the reading.

14.14.3.6 Repeat steps 14.14.3.4 and 14.14.3.5 until the aperture reading is within the range 0±20.

14.14.3.7 Replace the Allen wrench in the front cover of the instrument.

14.14.3.8 Replace the front cover on the instrument. Make sure that the cover is seated firmly on the instrument and that the cover is flush with the instrument.

14.14.4 End the APERTEST macro by pressing <F8>. The macro will take one last reading of the aperture and print that value out on the printer.

14.15 Enter the sample identifications into the computer.

14.15.1 Go to the main menu by pressing <F1>.

14.15.2 Select the **Autosampler** screen by press **A**.

14.15.3 Enter the **Rack entry** screen by pressing **R**.

14.15.4 At the bottom of the screen, there will be a prompt for the rack name. The rack name is a combination of the month, day, analysis number of the day, and rack number of the analysis in a specific order. The format is:

MDDRA-N

Where:
M is the coded month
DD is the two digit date of the month

R is the letter R. It stands for Rack.

A is the numeric representation of the number of separate analyses that have been performed that day. An analysis is defined as a set of samples that are digested together. For example, if a water digestion is analyzed first thing in the day, then A would be 1. If a soil digestion is then analyzed during that day, A would be 2.

N is the numeric representation of the number of separate racks that have been performed for a given analysis for that day. For example, if a soil digestion has 5 racks, the first rack would have an N value of 1, the second a value of 2, the third a value of 3, and so on.

Type in the rack name in the space provided and press <Enter>. If the rack name does not exist, the computer will ask if it should create a new rack name. Answer 'Y' and press <Enter>. Place the rack name on the autosampler ID/WT sheet in the space provided.

14.15.5 Press the <Insert> key once to begin editing the contents of the rack.

14.15.6 The first column, labeled 'cup', contains the autosampler position number that corresponds to the ID/WT sheet used for setting up the autosampler tray. This field may not be edited.

14.15.7 The second column, labeled 'Id', will contain the sample identification that is located on the ID/WT sheet. The number or identification put in this column will be the LIMS sample number for the sample.

14.15.8 The third column, called 'Extended Id', while not commonly used, may be used to further identify the sample.

14.15.9 The third and fourth columns on the computer screen will contain the sample digestion weight and volume for the sample. For example, if 0.1 g of sample was digested, then the dilution factor would be 0.1 in the weight column and 100 in the volume column (0.1g was digested into 100 ml final solution). If a subsequent

dilution is performed on the sample, then the final volume is multiplied by that dilution factor. For example, if in the above example, the final solution is then diluted 10X, then the final volume would be 1000. Please see the metals lab supervisor for further instruction on this topic.

14.15.10 There are a total of 44 positions for each tray. To go down to the next page of autosampler positions, press <Page Down>. To go up to the previous page, press <Page Up>.

14.15.11 When the ID/WT sheet is completely entered into the computer for a rack, press 'E' to exit.

14.15.12 Repeat steps 14.15.4 through 14.15.11 for the remaining racks.

14.16 Start the MERCURY macro.

14.16.1 Press <F2> (Macro).

14.16.2 Type in MERCURY in the space provided, then press <Enter>.

14.17 At this point the MERCURY macro should start initializing the system. The macro will ask the user a series of questions to determine the state of the instrument. If the macro fails to start or if there are any problems from this point on, contact the metals lab supervisor.

14.18 The computer will ask 'Would you like to do a warm start? <Y/N>'. The warm start macro is used to prepare the PS200 for operation from the short-term shut down. If the lamp has been shut off for more than 1 hour, and the instrument has been left on, enter "Y". See Section 9.9 for a description of the WARMSTRT macro.

14.19 The macro will then ask the user 'Would you like to test the aperture? <Y/N>'. If the aperture has already been tested, answer 'N', otherwise answer 'Y'. For a discussion on the APERTEST macro, see Section 14.4.

14.20 A data file name must now be entered. The data file name must be unique, otherwise, the data will be appended to another data file. The data file name is a combination of the month, day of the month, and analysis number of the day. The format is:

MDDRA

Where:
M is the coded month
DD is the two digit date of the month
R is the letter R. It stands for Rack.
A is the numeric representation of the number of separate analyses that have been performed that day. An analyses is defined as a set of samples that are digested together. For example, if a water digestion is analyzed first thing in the day, then A would be 1. If a soil digestion is then analyzed during that day, A would be 2.

Type in the data name in the space provided and press <Enter>. If the data name does not exist, the computer will ask if it should create a new one. Answer 'Y' and press <Enter>. Place the data file name on the autosampler ID/WT sheet in the space provided.

14.21 The instrument will now prompt for the rack name and the autosampler positions that should be analyzed.

14.21.1 At the prompt 'Enter rack name (ENTER for none)', place the name of the first rack to be analyzed. If there is no rack, press the <Enter> key to skip this entry.

14.21.2 Type in the first autosampler position that is to be analyzed. Again, press <Enter> if there are no samples in the first physical rack to analyze.

14.21.3 Type in the last autosampler position to be analyzed on the first rack. Press <Enter> if there are no samples to analyze on the rack.

14.21.4 At the prompt 'Enter rack name (ENTER for none)', place the name of the second rack to be analyzed. If there is no rack, press the <Enter> key to skip this entry.

14.21.5 Type in the first autosampler position that is to be analyzed. Again, press <Enter> if there are no samples in the second physical rack to analyze.

14.21.6 Type in the last autosampler position to be analyzed on the second rack. Press <Enter> if there are no samples to analyze on the rack.

14.22 When the printer is done printing, the macro will take the user to the check sample screen. The user must verify that the values that the macro is putting in are okay. If the values entered for the BLK, the ICV/CCV, or the LCS are not correct, change them when prompted. If the values need to be updated in the macro, contact the metals lab supervisor.

14.23 The macro will now pause so that the user may check the values that have been entered automatically by the macro. This pause also allows the user to correct any mistakes that were made when entering data at the macro prompts. This SOP will not attempt to describe all of the information that may be edited. The user may refer to the instrument manual or may contact the metals lab supervisor if there is something that must be changed before beginning the analysis. When all of the values are okay, press <F2> to resume the macro.

14.24 As an added protection, the macro will ask 'Are you sure you want to start? <Y/N>'. Answer with the appropriate response. The macro will not continue until 'Y' is entered.

14.25 The computer will ask 'Do standards need running? <Y/N>'. The instrument must be calibrated every 24 hours or every time that a new digestion batch is started. Answer the question appropriately. If the standards do need to be ran, see Section 13 for a detailed explanation of the calibration sequence.

14.26 When the standards are complete, the instrument will automatically analyze the ICB. The ICB should read back within ± 0.1 µg/L. If at any point in the decision tree below the ICB is within the limits, the macro will jump to step 14.27.

14.26.1 If the ICB does not meet the criteria, the macro will ask the user to place a new vial of the blank in autosampler position C1. Press any key to continue.

14.26.2 The instrument will then analyze the ICB again. If the ICB still does not meet the criteria, then the macro will autozero the instrument and rerun the ICB.

14.26.3 If the ICB does not read back within limits, the macro will essentially jump back to step 14.26.1. Please note that there is a possibility of an infinite loop occurring at this step. If the ICB refuses to work after 2 times through the decision tree, stop the macro and contact the metals lab supervisor for assistance.

14.27 When the ICB reads back within limits, the ICV will automatically be analyzed. The ICV is the midpoint calibration standard analyzed as a sample with the concentration being read from the analytical curve. The percent recovery for the ICV must be within the control limits of 90%-110% of the true value. If the ICV recovers within the control windows before the macro reslopes, then the macro will jump to Step 14.28.

14.27.1 If the ICV fails to recover within the control limits, the macro will ask the analyst to place a new vial of the ICV in autosampler position C2. When this is complete, press any key to continue the analysis.

14.27.2 If the ICV fails again, the macro will try to reslope the calibration based on an analysis of the midpoint standard. If the reslope fails, then the macro will rerun the standards and start again at Step 14.26.

14.27.3 If the reslope has a correlation coefficient greater than 0.995, then the macro will start again at Step 14.26.

14.28 The macro will now analyze an independent standard called the Laboratory Control Sample (LCS). The percent recovery must fall within the LIMS (Laboratory Information Management System) control limit for this type of QC. Please refer to the current LIMS control limits for the windows.

14.28.1 If the LCS does not fall within established control limits, the macro will ask the user to place another vial of the LCS in autosampler position C3. Press <Enter> to continue.

14.28.2 If the LCS still does not meet the criteria, then the macro will ask the user to prepare another LCS and to place the vial for the new LCS in autosampler position C3. Press any key to continue.

14.28.3 If the LCS still does not work, the macro will essentially begin again at Step 14.28.1. Please note that there is a possibility of an infinite loop occurring at this step. If the LCS does not work after 2 times through the decision tree, stop the macro and contact the metals lab supervisor for assistance.

14.29 The autosampler will now analyze 22 sample vials. If there are less than 22 vials left on the tray, then the autosampler will only analyze those samples. If the user needs to pause at any time during the analysis, press <F8>. The autosampler will stop after the current sample and wait until the <F8> key is pressed again. If the user presses the <F10> key, then the macro will stop immediately. The only way to resume a stopped analysis is to go to step 14.16 and restart the MERCURY macro.

14.30 When the autosampler has completed the analysis of the samples, the instrument will automatically analyze the CCB. The CCB should read back within ± 0.1 µg/L. If at any point in the decision tree below the CCB is within the limits, the macro will jump to step 14.31.

14.30.1 If the CCB does not meet the criteria, the macro will ask the user to place a new vial of the blank in autosampler position C1. Press any key to continue.

14.30.2 The instrument will then analyze the CCB again. If the CCB still does not meet the criteria, then the macro will autozero the instrument and rerun the CCB.

14.30.3 If the CCB does not read back within limits, the macro will essentially jump back to step 14.30.1. Please note that there is a possibility of an infinite loop occurring at this step. If the ICB refuses to work after 2 times through the decision tree, stop the macro and contact the metals lab supervisor for assistance.

14.31 When the CCB reads back within limits, the CCV will automatically be analyzed. The CCV is the midpoint calibration standard analyzed as a sample with the concentration

being read from the analytical curve. The percent recovery for the CCV must be within the control limits of 80%-120% of the true value. If the CCV recovers within the control windows before the macro reslopes, then the macro will jump to Step 14.32.

14.31.1 If the CCV fails to recover within the control limits, the macro will ask the analyst to place a new vial of the CCV in autosampler position C2. When this is complete, press any key to continue the analysis.

14.31.2 If the CCV fails again, the macro will try to reslope the calibration based on an analysis of the midpoint standard. If the reslope fails, then the macro will rerun the standards, set the autosampler to rerun all samples since the last good ICV or CCV, and start again at Step 14.26.

14.31.3 If the reslope has a correlation coefficient greater than 0.995, then the macro will set the autosampler to rerun all samples since the last good ICV or CCV and start again at Step 14.26.

14.32 If the CCV recovers within control limits, then start again at Step 14.29 with the next set of samples to be analyzed. If there are no more samples to analyze, then go to Step 14.33.

14.33 Check over the raw data to make sure that all of the samples are within the calibration range of the instrument. If any samples are above the calibration range of the curve, mark the sample number and appropriate dilution factor on the ID/WT sheet to be analyzed with the other dilutions at the end of the run. Dilutions may be made on the sample only if the diluent used has the correct amount of reagents in it. Do not use ASTM Type II water for dilutions. Make up a Blank solution of reagent water and reagents to use as the diluent.

15.0 FLOW CHART

Not Applicable

16.0 CALCULATIONS/DATA HANDLING

16.1 The concentration of each sample is read directly from the computer printout. Dilution factors should be taken into account in the ID/WT file.

16.1.1 Assuming that the instrument did not correct a concentration due to analyst error, the following calculation would be used:

$C_{Final} = C_{Instr} * DF_{Digest} * DF_{Subseq}$,, where

C_{Final} is the final reported concentration of the analyte
C_{Instr} is the concentration as read from the instrument data printout
DF_{Digest} is the digestion dilution factor
DF_{Subseq} is any dilution done subsequent to the digestion dilution

16.2 All samples should be reported to the correct number of significant figures. The significant figure truncation should not be performed until all data calculation have taken place.

16.2.1 Solid samples must be reported in mg/kg.

16.2.1.1 For sample concentrations <100 mg/kg, report 2 significant figures.

16.2.1.1 For sample concentrations ≥100 mg/kg, report 3 significant figures.

16.2.1.1 For QC, always report one additional significant figure.

16.2.2 Aqueous samples must be reported in ug/L.

16.2.2.1 For sample concentrations <1 ppb, report 1 significant figure.

16.2.2.2 For sample concentrations ≥1 ppb and <100 ppb, report 2 significant figures.

16.2.2.3 For sample concentrations ≥100 ppb, report 3 significant figures.

16.2.2.4 For QC, always report one additional significant figure.

16.2.3 Extracted samples for all metals must be reported in mg/L.

16.2.3.1 For sample concentrations <0.001 mg/L, report 1 significant figure.

16.2.3.2 For sample concentrations ≥0.001 mg/L and <0.100 mg/L, report 2 significant figures.

16.2.3.3 For sample concentrations ≥0.100 mg/L, report 3 significant figures.

16.2.3.4 For QC, always report one additional significant figure.

17.0 DATA REPORTING

See appendices F, H, I, and J for data reporting.

18.0 QUALITY ASSURANCE

18.1 All quality control data should be maintained and available for easy reference or inspection.

18.2 A calibration curve must be prepared each day with a minimum of a reagent blank and three standards. The correlation coefficient must be ≥ 0.995. If this fails the problem must be corrected.

18.3 IDL and MDL studies must be performed on an annual basis.

18.3.1 To calculate an IDL, analyze ten cups of Blank solution in series. Calculate the standard deviation of a small population, and multiply this result by three. There is also a spreadsheet located on the computer network that will perform this calculation for you. Please see the metals lab supervisor if you need assistance in using this program.

18.3.2 To calculate an MDL, analyze ten cups of a low concentration standard, typically at a concentration at or near the detection limit. Calculate the standard deviation of a small population and multiply the result by the Student's T value for (n-1) degrees of freedom. If the concentration of the standard ran is greater than ten times the calculated MDL, and the concentration of the standard ran is above the detection limit reported to clients, the MDL must be performed at lower concentration. If the standard concentration is less than ten times the calculated MDL or if the standard used for the analysis was at or below the lowest reporting limit on LIMS, the MDL is acceptable. There is also a spreadsheet located on the computer network that will perform this calculation for you. Please see the metals lab supervisor if you need assistance in using this program.

18.4 Dilute and reanalyze all samples that have concentrations greater than the highest standard of the analytical curve.

18.5 Include a minimum of one laboratory blank per sample digestion batch to determine if contamination or any memory effects are occurring. This laboratory blank must be carried through the sample preparation procedure.

18.6 Analyze one spike and one matrix spike duplicate (MSD) at a frequency of at least 1 in 20. A spike and matrix spike duplicate are samples into which a calculated extra amount of metal is pipetted in. The spike measures the accuracy of the sample preparation method and the MSD checks the precision of the method.

18.7 **Standard addition:** The Method of Standard Additions (MSA) shall be used for all EP extracts. MSAs must also be performed on all TCLP extracts that have spike recoveries less than 50%. The standard-addition technique involves adding known amounts of standard to one or more aliquots of the process sample solution. This technique compensates for a sample constitute that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct additive interferences which cause a baseline shift. The simple version of this technique is the single addition method, in which identical aliquots of the sample solution, each of

volume V_X, are taken. To the first (labeled A) is added a small volume V_S of a standard solution of concentration C_S. To the second (labeled B) is added the same volume V_S of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration C_X is calculated:

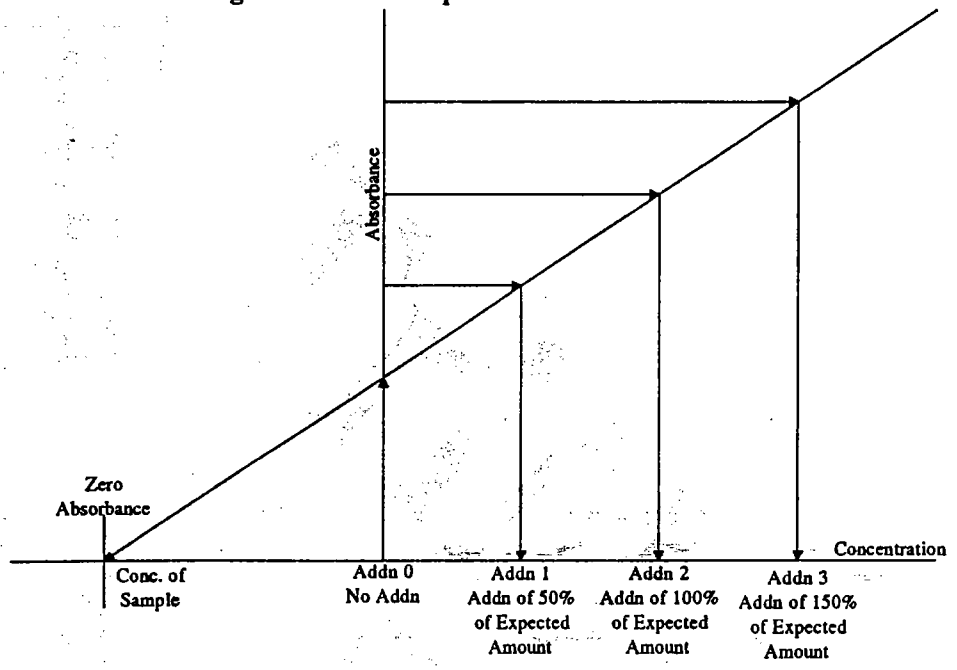
$$C_X = \frac{S_B * V_S * C_S}{(S_A - S_B) * V_X}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_S and C_S should be chosen so that S_A is roughly twice S_B on the average. It is best if V_S is made much less than V_X, and thus C_S is much greater than C_X, to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure. For the results of this technique to be valid, the following limitations must be taken into consideration:

1. The analytical curve must be linear and the correlation coefficient must be ≥ 0.995.
2. The chemical form of the analyte added must respond the same way as the analyte in the sample.
3. The interference effect must be constant over the working range of concern.
4. The signal must be corrected for any additive interference.

The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 1.

Figure 1 An Example of an MSA curve



18.8 Check the instrument standardization by analyzing appropriate quality control check standards as follows.

NOTE: Steps 18.8.2 through 18.8.6 must be performed every time the instrument is calibrated.

18.8.1 Calibrate the instrument using at least three standards and a blank.

18.8.2 An ICB (Initial Calibration Blank) is analyzed after the calibration. The results of this blank are to be within ± 0.1 µg/L. If the blank is not within ± 0.1 µg/L, correct the problem, and restart the analysis from the beginning.

18.8.3 An ICV (Initial Calibration Verification) is placed after the ICB. Percent recovery limits are 90% to 110% of the concentration. If the percent recovery for

this standard is not within the acceptance limits, correct the problem, recalibrate if necessary, and restart the analysis.

18.8.4 An LCS (Laboratory Check Standard), which must come from a different primary source than the calibration standards, is placed after the ICV in each run. Percent recovery limits are 80% to 120% of the concentration unless control limits have been established. If the percent recovery for this standard is not within the acceptance limits, correct the problem. If the instrument had to be recalibrated, restart the analysis from the beginning.

18.8.5 Verify the calibration after every 10 samples.

18.8.5.1 A CCV (Continuing Calibration Verification) is analyzed after every 10 samples, or sooner. Percent recovery limits are 80% to 120% of the concentration. If the percent recovery for this standard is not within the acceptance limits, correct the problem. When the problem has been corrected, analyze a CCV. If the CCV is within control limits, restart the analysis from the last good ICV or CCV. If the CCV is not within the control limits, repeat steps 18.8.1 through 18.8.4, and then reanalyze all samples since the last good ICV or CCV.

18.8.5.2 A CCB (Continuing Calibration Blank) is analyzed after the CCV. The results of this blank are to be within ± 0.1 µg/L. If the blank is not within ± 0.1 µg/L, correct the problem, and restart the analysis from the last good CCB or ICB.

18.8.6 At the end of each run, verify the calibration.

18.8.6.1 A CCV (Continuing Calibration Verification) is analyzed after every 10 samples, or sooner. Percent recovery limits are 80% to 120% of the concentration. If the percent recovery for this standard is not within the acceptance limits, correct the problem. When the problem has been corrected, analyze a CCV. If the CCV is within control limits, restart the analysis from the last good ICV or CCV. If the CCV is not within the

control limits, repeat steps 18.8.1 through 18.8.4, and then reanalyze all samples since the last good ICV or CCV.

18.8.6.2 A CCB (Continuing Calibration Blank) is analyzed after the CCV. The results of this blank are to be within ± 0.1 µg/L. If the blank is not within ± 0.1 µg/L, correct the problem, and restart the analysis from the last good CCB or ICB.

18.9 A new analytical curve must be ran every 24 hours.

18.10 If the % RSD of the replicates for a sample is greater than 20% and the concentration of the sample is greater than the reporting limit, reanalyze the sample once. If the % RSD is again greater than 20% on a sample, qualify the data with data Qualifier number 10.

19.0 ANALYST CERTIFICATION

See Appendix E

20.0 REFERENCES

20.1 U.S. Environmental Protection Agency. Mercury in Solid or Semisolid Waste (Manual Cold Vapor Technique) Method 7471A, SW-846 Test Methods for Evaluating Solid Waste, 3rd Edition, Revision 0, September 1994.

20.2 U.S. Environmental Protection Agency. Determination of Mercury in Sediments by Cold Vapor Atomic Absorption Spectrometry Method 245.5, revision 2.3, EPA-600/4-91/010, June 1991.

20.3 U.S. Environmental Protection Agency. Mercury (Automated Cold Vapor Technique) Method 245.2 1974, EPA-600/4-79-020, revised March 1983.

20.4 Gaskill, A., Compilation and Evaluation of RCRA Method Performance Data, Work Assignment No. 2, EPA Contract No. 68-01-7075, September 1986.

Approved By: ST 1-30-96 QA/QC Supervisor

Approved By: BQD 1-30-96 Area Supervisor

20.5 Leeman Labs PS Series ICP/Eschelle Spectrometers Reference Manual Rev. 1.0, May 1990.

21.0 APPENDICES

See attached pages.

Approved By: ST 1-30-96 QA/QC Supervisor

Approved By: BJD 1-30-96 Area Supervisor

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

MERCURY ATOMIC ABSORPTION FOR THE ANALYSIS OF LIQUID WASTE: AUTOMATED COLD VAPOR TECHNIQUE

MODIFIED METHODS 7470 and 245.1

APPROVALS:

Inorganic Manager: _____ Date: 12/4/98
Douglas E. Kriscunas

QA Manager: _____ Date: 12/4/98
Rick D. Wilburn

Laboratory Manager: _____ Date: 12/4/98
Douglas E. Kriscunas

Procedure Number: GR-01-123
Revision Number: 3.0

Date Initiated: 5/6/93
Effective Date: 12/4/98

Date Revised: 12/4/1998
Pages Revised: All

By: Dave Johnson

Total Number of Pages: 32

1.0 SCOPE AND APPLICATION

1.1 Method 7470/245.1 is a combination of USEPA Methods 7470, 245.1. This method may be used for the determination of total mercury (inorganic and organic) in mobility-procedure extracts, aqueous wastes, ground waters, drinking waters, surface waters, saline waters, domestic and industrial wastes. All samples analyzed by this method must undergo a procedure to destroy any organic substances in the sample prior to analysis.

2.0 PRINCIPAL METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update III, Revision 1, September, 1994, Method 7470A, "Mercury in Liquid Waste (Manual Cold-Vapor Technique)".

2.2 *Methods for the Determination of Metals in Environmental Samples*, Supplement I, May 1994, Revision 5.4, EMMC Version, "Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry", Method 245.1, Revision 3.0, May, 1994.

3.0 SUMMARY OF PROCEDURE

3.1 Prior to analysis, all samples, standards, and quality control samples must be prepared according to the procedure discussed in Section 12 of this method. This digestion procedure breaks down organo-mercury compounds into inorganic mercury. The inorganic mercury is then converted to the Hg^{+2} state for subsequent analysis

3.2 The prepared liquid sample with mercury in the divalent form (Hg^{+2}) enters the mercury analysis system and is mixed with stannous sulfate to form elemental mercury vapor (Hg^0) according to the following equation:

$$Hg^{+2} + Sn^{+2} \text{ -------> } Hg^0 + Sn^{+4}$$

3.3 The mixture flows into a liquid-gas separator where argon is introduced to carry the mercury through a drying tube containing magnesium perchlorate. The dry mercury vapor then enters one path of a heated double-path optical cell that has been optimized for fast response time. A mercury source powered by a constant current power supply delivers a stable source of emission at 253.7 nm. Absorbance by the mercury vapor is measured using a solid state detector with a wide dynamic range. The resulting signal is referenced to the simultaneous absorbance of the pure carrier gas flowing through the second optical path under identical conditions. The absorbances of the standards are plotted against the known concentration. Absorbance readings from unknown samples are then read from the standard curve and extrapolated to the concentration. The mercury vapor is then captured on activated carbon and the argon is vented up a hood to reduce the potential for contamination of other samples.

3.4 This SOP describes the current procedure that is in use at TriMatrix. Since this method uses the Leeman AP200 and PS200 to analyze aqueous samples, the following modifications have been implemented. The

amount of sample to be digested has been reduced from 100 ml to 8 ml due to sample vial size. All of the reagents in the method have been scaled back proportionally to the new sample volume of 8 ml. The new 245.1 method does not require that the standards be digested, but method 7470 still requires that the standards be digested. Since TriMatrix still digests all standards and quality control checks, TriMatrix will continue to use the control limits established in Method 7470 and the older 245.1 methods. The digestion of samples is accomplished in the Leeman AP200 digestion unit that adds all reagents, cooks the samples, reduces the samples, and purges the samples with argon. The analysis of samples utilizes the Leeman PS200 system that is a dedicated mercury analysis system. The PS200 is a flow-through system that reduces the mercury to the ground state, then sweeps the vapor into a heated flow cell.

3.5 The typical reporting limit for this method is 0.2 µg/L. The instrument detection limit for this procedure is 0.174 µg/L.

4.0 PARAMETER OR COMPOUND LIST

4.1 Mercury

5.0 REFERENCED SOPs

5.1 None referenced.

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Soil samples often contain mercury at low levels. When soils are mixed and ground, there exists the possibility that the liquid samples may become contaminated. Therefore, the preparation area for mercury must be kept free of dust and dirt to avoid contamination.

6.2 Certain volatile organic materials that absorb at this wavelength (253.7 nm) may also cause interference. If an interference is suspected, a preliminary run without reagents should determine if this type of interference is present.

6.3 The headspace at the top of the vial must be purged with argon prior to analysis. This step will sweep any chlorine away from the sample to reduce the potential from the chlorine interference.

6.4 The non-metals lab uses a product called Chrome-merge that contains mercury at fairly high concentrations to clean BOD bottles. Since some of the glassware used in this procedure is cleaned in the same area as the BOD bottles, care should be taken to minimize the potential of contamination from this source. If the glassware has become contaminated with the reagent, all suspected glassware must be thoroughly washed again prior to use.

6.5 If a mercury thermometer is broken anywhere in the laboratory, the metals lab will be notified. There always exists the possibility that sample vials may become contaminated from the mercury vapor from the broken thermometer. If contamination is suspected, the vials must be dumped and the samples redigested.

6.6 Mercury may be lost if the temperature of the water bath used to perform the digestion goes above 95°C. All water baths used for digestion must have their temperature monitored with an alcohol thermometer. The temperature of the water bath must be recorded in the mercury digestion logbook.

6.7 The reagents used in this procedure may become contaminated over time. If contamination is suspected, the reagent must be discarded and fresh reagent made.

7.0 SAFETY PRECAUTIONS

7.1 The analyst must comply with all standard operating procedures for health and safety as outlined in the TriMatrix Laboratory Safety Manual.

7.2 Concentrated acids are used in the preparation of standards and samples for analysis by cold vapor. Gloves and safety glasses must be worn at all times when handling concentrated acids. Gloves must also be worn when handling digested samples. Please refer to the MSDS for information on these or any other chemicals utilized in this procedure.

7.3 Check the exhaust hood over the instrument to be sure it is operating correctly. If the ventilation system is not working properly, immediately contact the metals lab supervisor. Under no circumstances should the cold vapor unit be used if the exhaust hood is not working.

7.4 UV protective glasses must be worn at all times in the metals laboratory. The mercury lamp emits UV radiation. Avoid looking directly at the lamp without some type of strong UV protection. Failure to follow this policy may cause very serious and immediate damage to the retina of the eye.

7.5 The cold vapor unit and the mercury digestion units use gaseous Ar. Although Ar in and of itself is not hazardous or flammable, it may cause suffocation through oxygen deprivation. It is therefore imperative that all sources of Ar be turned off with a valve when not in use. Since Ar is colorless and odorless, if you feel lightheaded, please evacuate the metals lab at once and notify the metals lab supervisor. Please refer to the MSDS for information on this or any other chemicals utilized in this procedure.

7.6 Mercury exists in many forms, and is toxic in a variety of ways. Mercury vapor is toxic if inhaled. Do not operate the instrument if it is not properly ventilated. Hg^{+2} is toxic if ingested. Always wash hands after handling the mercury standard.

7.7 No food or drink is allowed in the metals lab. Food or drink may become contaminated with acid or metals and may therefore be hazardous.

7.8 Wash hands before starting work. Chemicals may be present on the skin that may interfere with metals analysis. Wash hands before leaving the metals lab. Chemicals and acids may be on the skin that could eventually be ingested or passed on to a third party through casual contact.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 All sample containers must be pre-washed with detergents, acids and ASTM Type II water. Plastic and glass containers are both suitable, although glass is preferable since mercury may be adsorbed by certain plastics.

8.2 Aqueous samples must be preserved at the time of collection to pH <2 with HNO_3. The suggested maximum holding time for aqueous samples for mercury analysis is 28 days.

8.3 All samples and standards must be subjected to the digestion that is detailed in Section 12.

8.4 The minimum samples size for this test is 25 ml. If quality control is to be performed on the sample, 75 ml is the minimum sample volume required.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Laboratory analytical balance capable of weighing 0.1 mg.

9.2 Laboratory balance capable of weighing 0.1 g.

9.3 Eppendorf pipettors capable of delivering 50-1000 µl.

9.4 Class A volumetric flasks, various volumes.

9.5 Class A reusable pipettes, various volumes.

9.6 Oxford pipettes capable of 5 to 10 ml of solution.

9.7 Magnetic stir plate and stir bar.

9.8 Laboratory spatulas. The spatulas must be cleaned prior to use.

9.9 Glass alcohol thermometer. Mercury thermometers must never be used in this procedure. The thermometer must be able to reach 95°C.

9.10 15 ml sample vials, glass. The vials must be capable of fitting in the AP200 autosampler rack. Currently, all of the glass vials are blown specifically for use with the AP200 unit. If the vials are weak in any way, they will crack when subjected to the preparation procedure. Do not use vials that are cracked at the top since excess evaporation may occur during digestion.

9.11 Leeman Labs AP200 automated mercury preparation system. The computer-controlled instrument dispenses reagents to each mercury sample in accordance with methods 245.1 and 7470. The unit then heats the samples to 95°C in a water bath for 2 hours. The samples are then cooled and the excess permanganate is reduced by the instrument. After the samples have been purged with Ar gas, the samples are ready for analysis using the PS200.

9.12 Computer running Leeman AP 200 control software. An IBM compatible 80386SX16 or better computer with at least 1 MB RAM, a 40 MB hard disk, and a VGA color monitor is needed. The current software used by the instrument is AP200 version 2.043. The software version may be updated without notice if it performs at least as well as the older version. Major updates to the software that require the analyst to deviate from the stated procedure will require an update to this SOP.

9.13 Leeman Labs PS200 automated mercury system. The instrument has an attached autosampler that is capable of aspirating sample and reagents. A regulator is built into the instrument to deliver the Ar to the sample stream. The instrument is a double-beam instrument capable of measuring the change in intensity of a mercury lamp at 253.7 nm. The Ar used to purge the mercury vapor from the sample is used as the reference beam. To compensate for any aberrations in the optical path, the instrument has an adjustment screw to tune the path so that they react the same to the emission of the mercury lamp.

9.14 Computer running Leeman PS 200 control software. An IBM compatible 80386SX16 or better computer with at least 1 MB RAM, a 40 MB hard disk, and a VGA color monitor is needed. The current software used by the instrument is PS200 version 2.008. The software version may be updated without notice if it performs at least as well as the older version. Major updates to the software that require the analyst to deviate from the stated procedure will require an update to this SOP.

9.15 Epson printer or equivalent. The specific printer used must respond to Epson printing codes. The printer must be attached to the computer using an appropriate communication cable. Please refer to the Instrument manual for further printer specifications.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Check calibration on pipettors prior to use. Please refer to the Eppendorf pipettor SOP for calibration procedures and corrective actions.

10.2 Verify calibration of the balance. Please refer to the balance SOP for calibration procedures and corrective actions.

10.3 Inspect the Ar supply when the shift begins. If the liquid level of the Ar falls below the reorder mark, notify the person responsible for ordering gasses or the metals lab supervisor.

10.4 Inspect the waste container on the floor by the instrument every shift. If the jug is full, properly dispose of the liquid.

10.5 The reagent valves on the AP200 must be checked and replaced periodically. Inspect all of the reagent valves prior to use. If the valve appears to be leaking, replace the valve. Please refer to the AP200 users manual for instructions on changing the reagent valve. Note the replacement in the instrument maintenance logbook.

10.6 Since the AP200 operates in a very corrosive atmosphere, the autosampler needs periodic maintenance.

10.6.1 Prior to the start of each batch of samples, the following steps should be performed.

10.6.1.1 Wet the rails with isopropyl alcohol.

10.6.1.2 Move the tip assembly back and forth slowly along the rails to pick up the alcohol.

10.6.1.3 Wipe the rails dry with a clean soft cloth.

10.6.1.4 Place two drops of light machine oil on both rails.

10.6.1.5 Move the tip assembly back and forth slowly along the rails to pick up the oil.

10.6.1.6 Wipe the rails lightly to remove any excess oil.

10.6.1.7 Note the maintenance in the instrument maintenance logbook.

10.6.2 If the autosampler has been exposed to concentrated acid or if the autosampler jams during a digestion batch, perform the following steps.

10.6.2.1 Wet the rails and the entire tip assembly with water to dilute the acid and decontaminate all exposed surfaces.

10.6.2.2 Move the tip assembly back and forth slowly along the rails to get water into the bearings

10.6.2.3 Repeat Steps 9.6.2.1 through 9.6.2.2 until all of the acid has been removed from the assembly.

10.6.2.4 Perform Steps 9.6.1.1 through 9.6.1.7

10.7 Inspect the autosampler probe on the AP200 prior to each use. The probe should not be bent and should be able to enter each of the sample vials without obstruction. If the probe is bent, replace it with a new one. Note the change in the instrument maintenance logbook.

10.8 Inspect the printing quality of the printer. If the print is hard to read or light in appearance, replace the printer cartridge with the appropriate replacement.

10.9 Check all tubing on the PS200 for flattened areas, leaks, and clogs before use. If any problems are found, change the tubing and note the change in the instrument maintenance logbook.

10.10 Prior to analysis, rinse the entire PS200 system with 1:3 HCl for about ½ hour followed by ASTM Type II H_2O for another ½ hour.

10.11 The aperture on the PS200 must be checked before the analysis has begun. See the analytical method for a discussion on how the aperture is tested.

10.12 The PS200 instrument must be given the appropriate amount of time to warm up before use. If the instrument has been shut down in 'Overnight' mode, it is highly recommended that the 'WARMSTRT' macro be invoked to warm the instrument up. If the instrument has been shut down for an extended period of time, the 'COLDSTRT' macro should be used.

10.12.1 The WARMSTRT macro asks a series of questions to the user to ensure that everything is in place and is in working order. The instrument then warms up for 20 minutes to condition the tubing and stabilize the lamp. While answering the questions, if the user enters an invalid response, the macro will not continue. For example, one of the questions is 'Is the lamp on? <Y/N>'. If the user enters anything but 'Y', the macro will not continue. When the macro has completed the warm-up period, the message 'System Ready' is displayed on the screen. If the macro was called from another macro, such as the MERCURY macro, then control is given back to the calling macro and the calling macro will resume at the step after the call to the macro.

10.12.2 The COLDSTRT macro is similar to the WARMSTRT macro, but the warm-up period is much longer since it will be invoked only if the instrument has been shut down for an extended period of time. Again, the questions are programmed such that only certain responses will allow the macro to continue. The warm up time for this macro is 2½ hours. When the macro has completed the warm-up period the message 'Operation Complete' is displayed on the screen. If the macro was called from another macro, such as the MERCURY macro, then control is given back to the calling macro and the calling macro will resume at the step after the call to the macro.

10.13 Replace the activated charcoal in the gas exhaust line annually. Note the change in the instrument maintenance logbook.

11.0 CHEMICALS AND REAGENTS

11.1 Acids used in the preparation of standards and for sample processing must be reagent grade or better. Redistilled acids may be used if it has been demonstrated that the acid is free from contamination.

11.1.1 Concentrated hydrochloric acid, trace metal grade.

11.1.2 Concentrated nitric acid, trace metal grade

11.1.3 Concentrated sulfuric acid, trace metals grade.

11.2 **ASTM Type II water** (ASTM D1193). Deionized water is fed into an all glass distillation unit. The resulting distillate is immediately placed into a plastic container. Impurities are measured by the Inorganic group at TriMatrix. This is the only water acceptable for use in the Metals lab for dilutions or standard preparation.

11.3 **Standard stock solutions** are purchased primarily from Inorganic Ventures. All stock solutions are ICP grade single-element solutions at concentrations of 1000 or 10000 ppm.

11.4 Argon gas supply: Welding grade or better. Currently, this is plumbed from a liquid Ar tank located outside of the building. Ar is used as the carrier gas for analysis.

11.5 Potassium Persulfate, 5% solution (W/V). In a plastic 3L-glass container, place 150 g $K_2S_2O_8$ crystals. Add 3 L ASTM Type II water to the container. Place a stir bar in the solution and stir on a stir plate until all of the crystals have dissolved. This solution will take several hours to prepare. Keep the container closed when not in use to avoid contamination of the reagent. This reagent will be made fresh weekly, or sooner, if a precipitate has formed.

11.6 Potassium Permanganate, 5% solution (W/V). In a plastic 3-liter container, place 150 g $KMnO_4$ crystals. Add 3 L ASTM Type II water to the container. Place a stir bar in the solution and stir on a stir plate until all of the crystals have dissolved. Since this solution is nearly a saturated solution, this solution may take several hours to prepare. To check if the crystals have dissolved, a glass pipette may be used to probe the bottom of the container for excess crystals. If any of the solution is spilled, immediately clean up the spill with water. If a brownish stain remains, it may be removed from surfaces with hydroxylamine hydrochloride. If the solution touches exposed skin, the skin will turn brown. The brown discoloration is not harmful as long as the excess permanganate is washed off. The stain will wear off over time. Potassium permanganate will collect mercury if left exposed to the atmosphere. The container must be covered when not in use and should be stored in a dark cabinet to reduce the potential for auto-catalytic reduction.

11.7 Sodium Chloride Hydroxylamine Hydrochloride, 12% solution (W/V). In a plastic 3-liter container, place 360 g of sodium chloride and 360 g of hydroxylamine hydrochloride. Add 3 L of ASTM Type II water and a stir bar. Place the container on a stir plate and stir until all of the crystals have dissolved. This reagent must be prepared fresh daily.

11.8 Sulfuric Acid 0.5N. Rinse a clean 1 liter volumetric flask several times with ASTM Type II water. Place about 500 ml ASTM Type II water in the flask. Carefully add 14 ml concentrated sulfuric acid to the water. Carefully swirl the flask to mix the solution. Dilute to the mark with ASTM Type II water. Mix well.

11.9 Stannous Sulfate, 10% solution (W/V). Rinse a clean 500 ml volumetric flask several times with ASTM Type II water. Place about 250 ml 0.5 N H_2SO_4 in the flask. Add 50 g stannous sulfate and dilute the flask to volume with 0.5 N H_2SO_4. Mix well. When the crystals have gone into solution, filter through a 0.45 µm filter by using a vacuum apparatus and a side arm filtering flask. **NOTE:** This mixture is a suspension and must be stirred continuously during use, even after filtration.

12.0 STANDARDS PREPARATION

12.1 All standards are single-element standards. All standards are good for 1 day.

12.2 Preparation of the 10,000 ppb standard.

12.2.1 Rinse a clean 100 ml volumetric flask with ASTM Type II water several times.

12.2.2 Place about 20 ml of ASTM Type II water in the flask.

12.2.3 Add 0.5 ml concentrated trace metal grade HNO_3 to the flask and swirl.

12.2.4 Pipette 1 ml 1000 ppm stock standard solution into the volumetric flask.

12.2.5 Dilute the flask to the mark and mix well.

12.2.6 Update the stock standard logbook.

12.2.7 Transfer the standard number from the standard logbook to the volumetric flask.

12.3 Preparation of the 100 ppb standard.

12.3.1 Rinse a clean 100 ml volumetric flask with ASTM Type II water several times.

12.3.2 Place about 20 ml of ASTM Type II water in the flask.

12.3.3 Add 0.5 ml concentrated trace metal grade HNO_3 to the flask and swirl.

12.3.4 Pipette 1 ml 10,000 ppb stock standard solution prepared in Step 11.2 into the volumetric flask.

12.3.5 Dilute the flask to the mark and mix well.

12.3.6 Update the stock standard logbook.

12.3.7 Transfer the standard number from the standard logbook to the volumetric flask.

12.4 Preparation of working standards.

12.4.1 Six calibration standards are prepared for use in the initial calibration curve. The six standards include a blank, a low level standard at the reporting limit (nominally 0.2 µg/L), 3 mid level standards, and a high standard near the upper linear range point (typically 5 µg/L). The standards are prepared as follows. Rinse six clean 100 ml volumetric flasks several times with ASTM Type II water.

12.4.2 Label the flasks as follows:

5 µg/L Hg-100 ml volumetric flask
2 µg/L Hg-100 ml volumetric flask
1 µg/L Hg-100 ml volumetric flask
0.5 µg/L Hg-100 ml volumetric flask
0.2 µg/L Hg-100 ml volumetric flask
Hg blank-100 ml volumetric flask

12.4.3 Add approximately 20 ml ASTM Type II water to each flask.

12.4.4 Add 0.5 ml concentrated trace metal grade HNO_3 to each flask and swirl.

12.4.5 Pipet 5 ml of the 100 µg/L Hg standard and transfer it to the 100 ml flask labeled 5 µg/L Hg. Dilute to volume and mix.

12.4.6 Pipet 2 ml of the 100 µg/L Hg standard and transfer it to the 100 ml flask labeled 2 µg/L Hg. Dilute to volume and mix.

12.4.7 Pipet 1 ml of the 100 µg/L Hg standard and transfer it to the 100 ml flask labeled 1 µg/L Hg. Dilute to volume and mix.

12.4.8 Pipet 0.5 ml of the 100 µg/L Hg standard and transfer it to the 100 ml flask labeled 0.5 µg/L Hg. Dilute to volume and mix.

12.4.9 Pipet 0.2 ml of the 5 µg/L Hg standard and transfer it to the 100 ml flask labeled 0.2 µg/L Hg. Dilute to volume and mix.

12.4.10 Dilute the flask labeled Hg blank to volume and mix well.

12.4.11 All working standards must be digested within 8 hours of preparation.

12.4.12 Complete the standards log book (see Appendix J).

12.4.13 Transfer the information from the standard logbook to the standard flasks and the ID/Wt sheet used for the analysis.

13.0 SAMPLE PREPARATION

13.1 All samples, standards, and quality control check samples must be digested before analysis.

13.2 Fill out the ID/WT sheet for the analysis. Each line on the ID/WT sheet represents a discreet autosampler position. If a sample needs a SPK and an MSD, then the sample will require three autosampler positions. If a dilution is to be performed on the sample, place the dilution factor in the column provided. NOTE: Continuing calibration verifications and blanks do not need to be placed in between samples, the PS200 will automatically go back to a special vial for these samples.

13.3 Load the autosampler tray with the samples to be prepared.

13.3.1 The first rack, the standards rack, contains 14 autosampler positions. Place seven 30 ml vials in autosampler positions S1 through S7. Autosampler positions S1 through S6 are reserved for standard solutions. S7 will always contain an empty vial for the unit to rinse into. Place 24 ml

of the standard solutions into the appropriate autosampler vials. Please refer to Table 1 for autosampler positions for the standards.

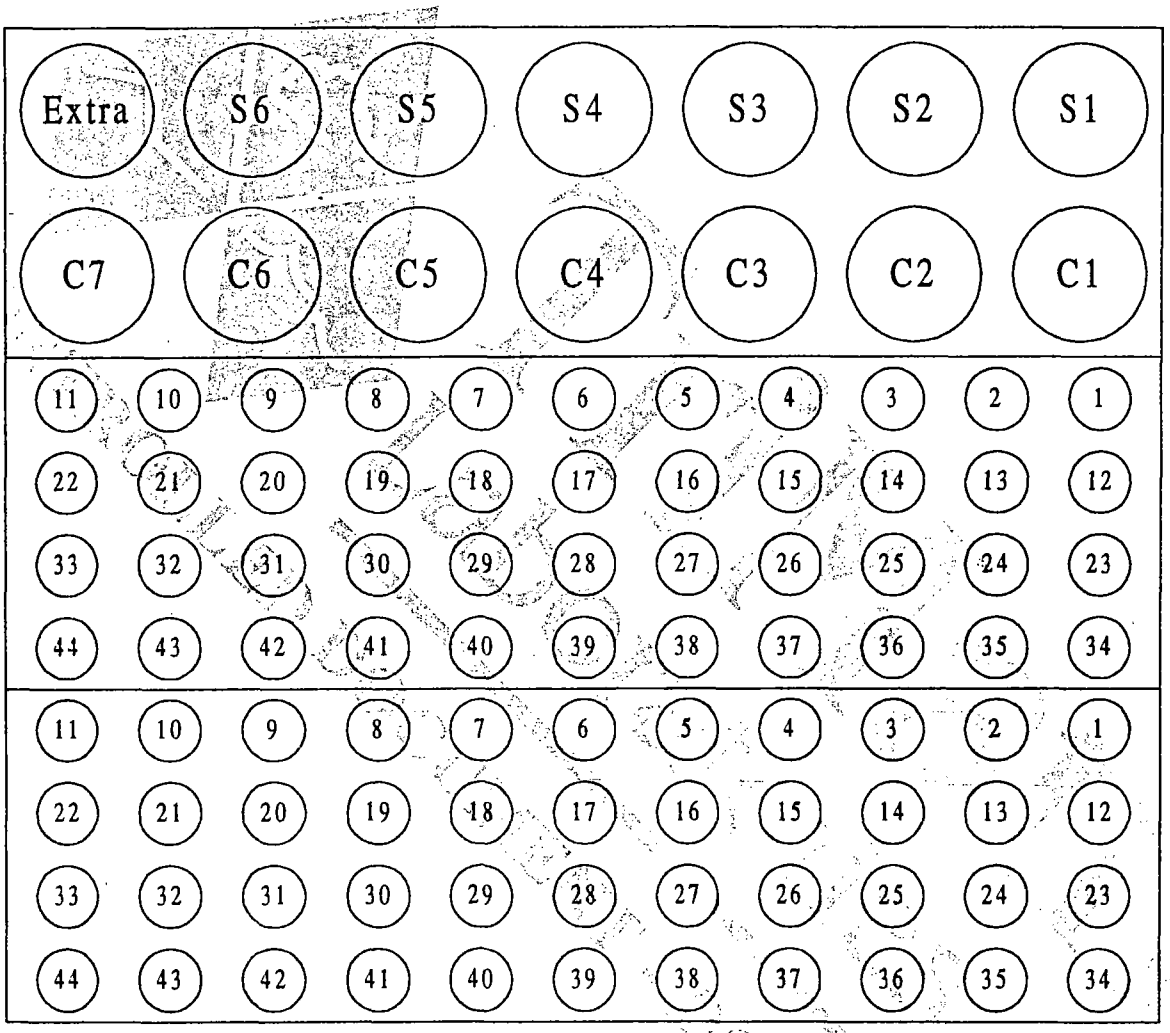
13.3.2 Place three 30 ml vials in autosampler positions C1 through C3. Place 24 ml of the quality control solutions into the appropriate autosampler vials. Please refer to Table 1 for autosampler positions for the quality control samples.

Table 1. Autosampler Positions for Standards and QCs

Autosampler Position	Standard Concentration (ppb)/Name
Rack 0, Position 1 (S1)	0 (Blank)
Rack 0, Position 2 (S2)	0.2
Rack 0, Position 3 (S3)	0.5
Rack 0, Position 4 (S4)	1.0
Rack 0, Position 5 (S5)	2.0
Rack 0, Position 6 (S6)	5.0
Rack 0, Position 8 (C1)	0 (Blank)
Rack 0, Position 9 (C2)	2.0 (ICV/CCV)
Rack 0, Position 10 (C3)	LCS
Rack 0, Position 11 (C4)	LCS

13.3.3 Load the samples into the autosampler trays. Place a vial containing 8 ml of the appropriate sample into its assigned position. While loading the tray, frequently refer to the ID/WT sheet to ensure that the sample is going into the correct autosampler position. Please refer to Figure 1 to determine how the autosampler rack is numbered.

Figure 1. Autosampler Rack Setup for the AP200 and PS200



13.4 Add concentrated H_2SO_4 to all standards and samples.

13.4.1 To all standards add 1.25 ml

13.4.2 To all samples add 0.4 ml

13.5 Add concentrated HNO_3 to all standards and samples.

13.5.1 To all standards add 0.625 ml

13.5.2 To all samples add 0.2 ml

13.6 Add $KMnO_4$ to all standards and samples and wait at least 15 minutes before proceeding to 13.7.

13.6.1 To all standards add 7.5 ml

13.6.2 To all samples add 2.5 ml

13.7 Add concentrated K_2SO_4 to all standards and samples.

13.7.1 To all standards add 2 ml

13.7.2 To all samples add 0.66 ml

13.8 Heat water in a roasting pan to 95°C.

13.9 Insert the standard vials and sample vials into the water bath and place tin foil around the sample racks to keep the heat in.

13.10 Once the water bath reaches 95°C again, set and start a 2 hour timer. After 2 hours, remove the samples from the water bath and allow to cool.

13.11 Once the standards and samples have cooled, add $NH_4OH{\cdot}HCl$.

13.11.1 To all standards add 3.0 ml

13.11.2 To all samples add 1.0 ml

13.12 Print and fill out a LIMS pretreatment benchsheet. See Appendix F.

13.13 Pretreat the samples on LIMS. See Appendix C.

13.14 Fill out the mercury digestion logbook. See Appendix D.

14.0 CALIBRATION PROCEDURES

14.1 Calibration of the instrument must be performed every 24 hours or for every batch of samples that were prepared in the same manner, whichever is more frequent. For example, if a soil calibration was performed at 8 AM and a water run is to be performed at 5 PM of the same day, a new calibration curve must be performed with standards prepared with the samples to be analyzed. A blank and five standards are run to comprise the initial calibration curve, as described in section 11.4. A minimum of a blank and three standards are required to be included in the initial calibration curve.

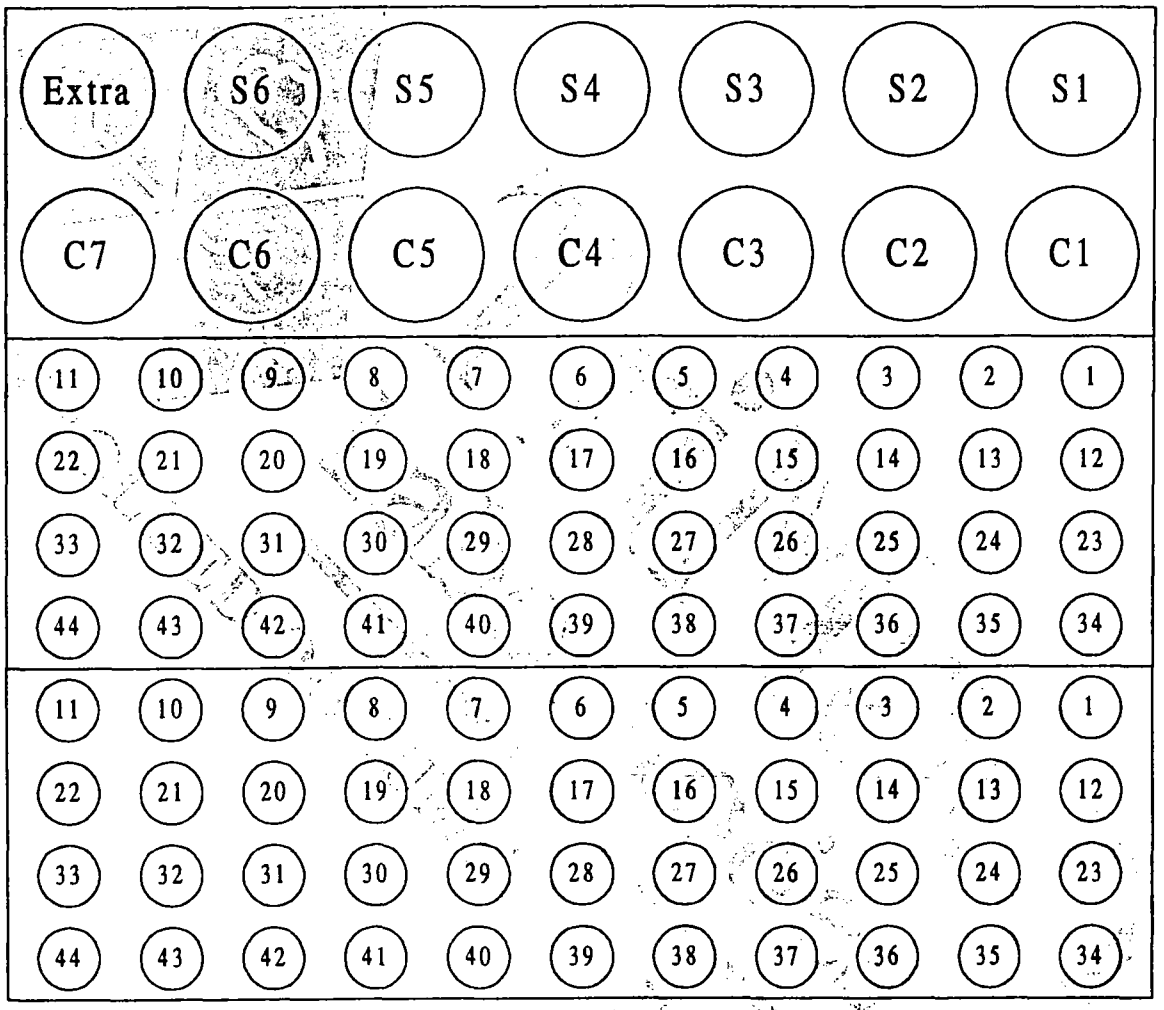
14.2 Start the instrument software as described in Section 14. Perform a COLDSTRT or a WARMSTRT, whichever is needed.

14.3 Load the autosampler tray with the appropriate standards. See Table 2 for autosampler positions. Rack 0 is the rack that is furthest away from the front of the instrument. See Figure 2 for autosampler position designation.

Table 2. Autosampler Positions for Standards

Autosampler Position	Standard Concentration (ppb)
Rack 0, Position 1 (S1)	0 (Blank)
Rack 0, Position 2 (S2)	0.2
Rack 0, Position 3 (S3)	0.5
Rack 0, Position 4 (S4)	1.0
Rack 0, Position 5 (S5)	2.0
Rack 0, Position 6 (S6)	5.0

Figure 2. Autosampler Position Designation



14.4 There are currently three ways to calibrate the instrument. Although the three methods are different in the keystrokes used, they are essentially performing the same task.

14.4.1 Method 1: Allow the MERCURY macro to run the standards unattended. This method will automatically initialize all parameters, run the standards, check the QC, run samples, and run periodic QC.

14.4.1.1 Start the MERCURY macro. When the PS200 software is booted up, the MERCURY macro is automatically invoked. If, for some reason the macro is stopped, the macro can be restarted by pressing <F2> then typing in MERCURY and pressing <Enter>.

14.4.1.2 Follow the direction outlined in Section 14 until the software asks if standards should be ran. Answer 'Y' to this question.

14.4.1.3 The instrument will automatically run the standards and plot the calibration curve when finished. Skip to Step 13.5.

14.4.2 Method 2: Use the RUNSTDS macro to run the standards unattended. This method will run the standards unattended and then stop. The user will have to either invoke the MERCURY macro again to restart the analysis in unattended mode or else the run will have to be performed manually.

14.4.2.1 Set up the instrument as described in Section 14. Perform a WARMSTRT or a COLDSTRT, if needed.

14.4.2.2 Start the macro by pressing <F2>, then type in RUNSTDS and press <Enter>.

14.4.2.3 The instrument will automatically run the standards and plot the calibration curve when finished. Skip to Step 13.5.

14.4.3 Method 3: Run each standard individually. This method needs constant operator attendance and should not be used if the entire curve is to be ran. The operator is responsible for checking all values before running the analysis if this method is used.

14.4.3.1 Press <F6> (Standard).

14.4.3.2 The software will ask for the standard number to run. Enter the standard number that should be analyzed. Do not enter the standard concentration, only the standard position. Refer to Figure 2 for the standard autosampler position.

14.4.3.3 Repeat steps 13.4.3.1 and 13.4.3.2 for the remaining standards that need to be ran.

14.4.3.4 When finished running the standards, go to the calibration page by pressing <F1> (Main Menu) then pressing <C> (Calibration) <L> (Line calibration). At this point a plot of the calibration should be displayed.

14.4.3.5 Press <A> to accept the calibration curve.

14.5 The correlation coefficient 'r' must be equal to or greater than 0.995. If the correlation coefficient is not equal to or greater than 0.995, up to 2 of the standard results may be deleted from the curve, excluding the blank and the low level standard run at the reporting limit. If the high level standard is one of the results deleted, the linear range of the curve has been decreased, and required sample dilutions will be adjusted accordingly. If the deletion of standards results does not solve the problem, corrective actions will be taken, including re-analysis of individual points, or the entire initial calibration curve.

14.5.1 Rerunning a point in the calibration curve.

14.5.1.1 Press <F6> (Standard).

14.5.1.2 The software will ask for the standard number to run. Enter the standard number that should be analyzed. Do not enter the standard concentration, only the standard position. Refer to Figure 2 for the standard autosampler position.

14.5.1.3 Repeat steps 13.5.1.1 and 13.5.1.2 for the remaining standards that need to be ran. **NOTE:** The software will not automatically re-calculate the correlation coefficient. The user must press <A> to re-calculate the coefficient.

14.5.2 Deleting a point in the curve that is not correct.

14.5.2.1 Since it is possible that there was a determinate error in the preparation of the standards, standard points may be dropped if they appear to significantly deviate from the curve. At least 3 standards and the blank must remain in the curve for the calibration curve to be valid.

14.5.2.2 Use the arrow keys to highlight the point that is to be deleted from the curve.

14.5.2.3 Press <Enter> to delete the point from the calibration curve.

14.5.2.4 Press <A> to re-calculate the calibration curve and correlation coefficient.

14.5.3 Add a previously deleted point back to the curve.

14.5.3.1 If a calibration point has been deleted by accident, or if it was deleted to check its affect on the remaining curve, the point may be added back without re-running the point. At least 3 standards and the blank must remain in the curve for the calibration curve to be valid.

14.5.3.2 Use the arrow keys to highlight the point that is to be added back to the curve.

14.5.3.3 Press <Enter> to add the point to the calibration curve.

14.5.3.4 Press <A> to re-calculate the calibration curve and correlation coefficient.

14.6 If the MERCURY macro was used to produce the analytical curve, press <F2> when the curve is acceptable. The software will double check that the correlation coefficient is correct, then print the analytical curve. The macro will then automatically start analyzing the quality control samples and samples. See Section 14 for a more in depth discussion on performing the analysis.

14.7 If the RUNSTDS macro was used to produce the analytical curve, press <F2> when the curve is acceptable. The software will double check that the correlation coefficient is correct, then print the analytical curve. The macro will then end. At this point, the quality control samples must be analyzed. If an unattended mode of operation is preferred, start the MERCURY macro by pressing <F2> and then

typing in MERCURY<Enter>. Performing the analysis manually is beyond the scope of this procedure. See Section 14 for a more in depth discussion on performing the analysis.

14.8 If the curve was constructed by manually running the standards, double check that the curve is accepted by pressing <A>. Check to ensure that the correlation coefficient is still 0.995 or greater. If the correlation is not at least 0.995, go back to step 13.5 and edit the calibration. If the correlation is okay, print the curve by pressing <F4> (Print Screen). At this point, the quality control samples must be analyzed. If an unattended mode of operation is preferred, start the MERCURY macro by pressing <F2> and then typing in MERCURY<Enter>. Performing the analysis manually is beyond the scope of this procedure. See Section 14 for a more in-depth discussion on performing the analysis.

15.0 ANALYTICAL PROCEDURE

15.1 This procedure assumes that the user has been introduced to the software either by reading the software manual or by experienced metals personnel. Advanced topics, such as how to replace the mercury source lamp are beyond the scope of this method. In this case, the user should reference the instrument manual.

15.2 Make sure that the argon supply is on. There is a valve behind the instrument that supplies the PS200 instrument. Make sure that the valve is open.

15.3 Turn on the instrument on if it is not already on. The power button for the instrument is the blue button on the front of the instrument. If the instrument fails to turn on, contact the metals lab supervisor.

15.4 Turn the computer and printer on if they are not turned on. If the computer fails to boot, contact the metals lab supervisor.

15.5 Turn the lamp on. The lamp power button is the green button on the front of the instrument. Allow the lamp to warm up for at least ½ hour before testing the aperture. If the lamp does not seem to be working, contact the metals lab supervisor.

15.6 The computer will automatically start the MERCURY macro. Stop the macro by pressing <F10> (Stop).

15.7 Place the stannous sulfate line (the line connected to the red/red tubing) and the sample line (the line connected to the black Viton tubing) in the wash vessel. Fill the wash vessel with 10% HCl solution.

15.8 Check the peristaltic tubing. If the tubing is blocked or badly worn, replace it with new tubing.

15.9 Clamp the tubing in place. Make sure that the peristaltic tubing is in a straight line over the pump rollers. Lift the red lever on the pump manifold to press the tubing against the rollers.

15.10 Start the pump. Go to the **Instrument** screen by pressing **I**. Press **O** to go to the **Operation** screen. Press **R O** to start the pump.

15.11 Turn on the Argon carrier gas. While in the **Instrument Operation** screen, press **G O** to turn on the carrier gas.

15.12 Check the peristaltic tubing to ensure that the pump is drawing liquid. Lift the sample line out of the rinse solution to see if the pump will pull air. Check the stannous sulfate line the same way. Watch the drain line right after the liquid/gas separator to see if bubbles are being drawn through the tubing. If none of the tubing is drawing, make sure that the pump is turning. If a single piece of tubing is not drawing, try to increase the tension to make the tubing draw by lifting the red lever on the pump manifold higher. If liquid is still not being drawn, try changing the tubing (See Section ####). If the pump still does not draw, a clog should be suspected. Please see the metals lab supervisor for help in finding and correcting the cause of the clog.

15.13 Fill the drying tube with drying agent. Currently, the metals lab is using Magnesium Perchlorate (Anhydrone) as the drying agent for mercury determinations.

15.13.1 Place a small amount of glass wool in one end of the drying tube. There should be just enough glass wool to keep the drying agent from falling out of the tube. Do not put too much glass wool in the end of the tube as this may restrict gas flow through the drying tube.

15.13.2 While wearing gloves, carefully fill the drying tube with fresh magnesium perchlorate. Fill the tube up to the neck of the tube. Gently tap the bottom of the tube on the counter to help the crystals settle into place. Do not over pack the tube as this will restrict the gas flow through the tube. Fill the tube up again within ¾ inch of the end of the tube.

15.13.3 Place a small amount of glass wool in the end of the drying tube. There should be just enough glass wool to keep the drying agent from falling out of the tube. Do not put too much glass wool in the end of the tube as this may restrict gas flow through the drying tube.

15.13.4 Check the O-rings on the drying tube endcaps for wear. Replace these O-rings if there is obvious wear.

15.13.5 Place the drying tube end caps on the end of the drying tube. Push the ends on until they stop. Tighten the nut on the endcaps until snug. Do not over tighten the endcaps.

15.13.6 Place the drying tube in its holder on the instrument.

15.14 If the lamp has been on for at least ½ hour, test the aperture.

15.14.1 Start the APERTEST macro.

15.14.1.1 Press <F2> (Macro).

15.14.1.2 Type in APERTEST, then press <Enter>.

15.14.2 The macro will now start to display some numbers on the screen. These numbers are the readings for the aperture and they represent the difference in intensity between the sample beam of the instrument and the reference beam.

15.14.3 The aperture reading must be 0±100, preferably 0±20. If the reading is not within specifications, adjust the aperture until the value is within range. Adjustment should be done as quickly as possible so that the temperature of the flow cell does not change significantly.

15.14.3.1 Remove the front cover from the instrument to reveal the flow cell. Push the cover up and then pull forward to remove.

15.14.3.2 Remove the Allen wrench from the front panel. The wrench is used to turn the aperture screws.

15.14.3.3 There are two screws on the flow cell at the right-hand end of the flow cell. Always adjust the screw that has been turned into the flow cell the furthest.

15.14.3.4 If the reading is negative, turn the screw clockwise until the reading is zero.
NOTE: In order to obtain an accurate reading, let go of the Allen wrench while taking the reading. Any pressure applied to the flow cell during the reading will change the reading.

15.14.3.5 If the reading is positive, turn the screw counter-clockwise until the reading is zero.
NOTE: In order to obtain an accurate reading, let go of the Allen wrench while taking the reading. Any pressure applied to the flow cell during the reading will change the reading.

15.14.3.6 Repeat steps 14.14.3.4 and 14.14.3.5 until the aperture reading is within the range 0±20.

15.14.3.7 Replace the Allen wrench in the front cover of the instrument.

15.14.3.8 Replace the front cover on the instrument. Make sure that the cover is seated firmly on the instrument and that the cover is flush with the instrument.

15.14.4 End the APERTEST macro by pressing <F8>. The macro will take one last reading of the aperture and print that value out on the printer.

15.15 Enter the sample identifications into the computer.

15.15.1 Go to the main menu by pressing <F1>.

15.15.2 Select the **Autosampler** screen by press **A**.

15.15.3 Enter the **Rack entry** screen by pressing **R**.

15.15.4 At the bottom of the screen, there will be a prompt for the rack name. The rack name is a combination of the month, day, analysis number of the day, and rack number of the analysis in a specific order. The format is:

MDDRA-N

Where:

M is the coded month

DD is the two digit date of the month

R is the letter R

A is the numeric representation of the number of separate analyses that have been performed that day. An analysis is defined as a set of samples that are digested together. For example, if a water digestion is analyzed first thing in the day, then A would be 1. If a soil digestion is then analyzed during that day, A would be 2.

N is the numeric representation of the number of separate racks that have been performed for a given analysis for that day. For example, if a soil digestion has 5 racks, the first rack would have an N value of 1, the second a value of 2, the third a value of 3, and so on.

For example, for the first rack of the first run analyzed on October 6, the rack name would be C06R1-1.

Type in the rack name in the space provided and press <Enter>. If the rack name does not exist, the computer will ask if it should create a new rack name. Answer 'Y' and press <Enter>. Place the rack name on the autosampler ID/WT sheet in the space provided.

15.15.5 Press the <Insert> key once to begin editing the contents of the rack.

15.15.6 The first column, labeled 'cup', contains the autosampler position number that corresponds to the ID/WT sheet used for setting up the autosampler tray. This field may not be edited.

15.15.7 The second column, labeled 'Id', will contain the sample identification that is located on the ID/WT sheet. The number or identification put in this column will be the LIMS sample number for the sample.

15.15.8 The third column, called 'Extended Id', while not commonly used, may be used to further identify the sample.

15.15.9 The third and fourth columns on the computer screen will contain the sample digestion weight and volume for the sample. For example, if 0.1 g of sample was digested, then the dilution factor would be 0.1 in the weight column and 100 in the volume column (0.1g was digested into 100 ml final solution). If a subsequent dilution is performed on the sample, then the final volume is multiplied by that dilution factor. For example, if in the above example, the final solution is then diluted 10X, then the final volume would be 1000. Please see the metals lab supervisor for further instruction on this topic.

15.15.10 There are a total of 44 positions for each tray. To go down to the next page of autosampler positions, press <Page Down>. To go up to the previous page, press <Page Up>.

15.15.11 When the ID/WT sheet is completely entered into the computer for a rack, press 'E' to exit.

15.15.12 Repeat steps 14.15.4 through 14.15.11 for the remaining racks.

15.16 Start the MERCURY macro.

15.16.1 Press <F2> (Macro).

15.16.2 Type in MERCURY in the space provided, then press <Enter>.

15.17 At this point the MERCURY macro should start initializing the system. The macro will ask the user a series of questions to determine the state of the instrument. If the macro fails to start or if there are any problems from this point on, contact the metals lab supervisor.

15.18 The computer will ask 'Would you like to do a warm start? <Y/N>'. The warm start macro is used to prepare the PS200 200 for operation from the short-term shut down. If the lamp has been shut off for more than 1 hour, and the instrument has been left on, enter "Y". See Section 9.9 for a description of the WARMSTRT macro.

15.19 The macro will then ask the user 'Would you like to test the aperture? <Y/N>'. If the aperture has already been tested, answer 'N', otherwise answer 'Y'. For a discussion on the APERTEST macro, see Section 14.4.

15.20 A data file name must now be entered. The data file name must be unique, otherwise, the data will be appended to another data file. The data file name is a combination of the month, day of the month, and analysis number of the day. The format is:

MDDRA

Where:

M is the coded month

DD is the two digit date of the month

R is the letter R

A is the numeric representation of the number of separate analyses that have been performed that day. An analyses is defined as a set of samples that are digested together. For example, if a water digestion is analyzed first thing in the day, then A would be 1. If a soil digestion is then analyzed during that day, A would be 2.

For example, for the first run analyzed on October 6, the data file name would be C06R1.

Type in the data name in the space provided and press <Enter>. If the data name does not exist, the computer will ask if it should create a new one. Answer 'Y' and press <Enter>. Place the data file name on the autosampler ID/WT sheet in the space provided.

15.21 The instrument will now prompt for the rack name and the autosampler positions that should be analyzed.

15.21.1 At the prompt 'Enter rack name (ENTER for none)', place the name of the first rack to be analyzed. If there is no rack, press the <Enter> key to skip this entry.

15.21.2 Type in the first autosampler position that is to be analyzed. Again, press <Enter> if there are no samples in the first physical rack to analyze.

15.21.3 Type in the last autosampler position to be analyzed on the first rack. Press <Enter> if there are no samples to analyze on the rack.

15.21.4 At the prompt 'Enter rack name (ENTER for none)', place the name of the second rack to be analyzed. If there is no rack, press the <Enter> key to skip this entry.

15.21.5 Type in the first autosampler position that is to be analyzed. Again, press <Enter> if there are no samples in the second physical rack to analyze.

15.21.6 Type in the last autosampler position to be analyzed on the second rack. Press <Enter> if there are no samples to analyze on the rack.

15.22 When the printer is done printing, the macro will take the user to the check sample screen. The user must verify that the values that the macro is putting in are okay. If the values entered for the BLK, the ICV/CCV, or the LCS are not correct, change them when prompted. If the values need to be updated in the macro, contact the metals lab supervisor.

15.23 The macro will now pause so that the user may check the values that have been entered automatically by the macro. This pause also allows the user to correct any mistakes that were made when entering data at the macro prompts. This SOP will not attempt to describe all of the information that may be edited. The user may refer to the instrument manual or may contact the metals lab supervisor if there is something that must be changed before beginning the analysis. When all of the values are okay, press <F2> to resume the macro.

15.24 As an added protection, the macro will ask 'Are you sure you want to start? <Y/N>'. Answer with the appropriate response. The macro will not continue until 'Y' is entered.

15.25 The computer will ask 'Do standards need running? <Y/N>'. The instrument must be calibrated every 24 hours or every time that a new digestion batch is started. Answer the question appropriately. If the standards do need to be ran, see Section 13 for a detailed explanation of the calibration sequence.

15.26 When the standards are complete, the instrument will automatically analyze the ICB. The amount found in the ICB must not be greater than the absolute value of the reporting limit. If at any point in the decision tree below the ICB is within the limits, the macro will jump to step 14.27.

15.26.1 If the ICB does not meet the criteria, the macro will ask the user to place a new vial of the blank in autosampler position C1. Press and key to continue.

15.26.2 The instrument will then analyze the new ICB. If the ICB still does not meet the criteria, then the macro will autozero the instrument and rerun the ICB.

15.26.3 If the ICB does not read back within limits, the macro will essentially jump back to step 14.26.1. Please note that there is a possibility of an infinite loop occurring at this step. If the analysis of the ICB still does not pass the criteria after the instrument has been autozeroed, stop the macro and contact the metals lab supervisor for assistance. Analysis cannot continue until an acceptable ICB has been analyzed.

15.27 When the ICB reads back within limits, the ICV will automatically be analyzed. The ICV is the midpoint calibration standard analyzed as a sample with the concentration being read from the analytical curve. The percent recovery for the ICV must be within the control limits of 80%-120% of the true value. If the ICV recovers within the control windows, the macro will jump to Step 14.28.

15.27.1 If the ICV fails to recover within the control limits, the macro will ask the analyst to place a new vial of the ICV in autosampler position C2. When this is complete, press any key to continue the analysis.

15.27.2 If the ICV fails again, corrective action measures must be implemented. If a mechanical failure can be found which has caused the out of control ICV, correct the problem and return to section 14.26, ICB analysis. If other corrective action procedures are required, such as gas flow adjustments or tubing replacement, the initial calibration sequence must be initiated again. Analysis may not begin until an acceptable ICV has been analyzed.

15.28 The macro will now analyze an independent standard called the Laboratory Control Sample (LCS). The LCS is a second source purchased standard, at a nominal concentration of 3.0 µg/L. The percent recovery must fall within the LIMS (Laboratory Information Management System) control limit for this type of QC. Please refer to the current LIMS control limits for the windows.

15.28.1 If the LCS does not fall within established control limits, the macro will ask the user to place another vial of the LCS in autosampler position C3. Press <Enter> to continue.

15.28.2 If the LCS still does not meet the criteria, then the macro will ask the user to prepare a new LCS and to place the vial for the new LCS in autosampler position C3. Press any key to continue.

15.28.3 If the LCS still does not work, stop the macro and contact the metals lab supervisor for assistance. Analysis may not begin until an acceptable LCS has been analyzed.

15.29 The autosampler will now analyze 10 sample vials. If there are less than 10 vials left on the tray, then the autosampler will only analyze those samples. If the user needs to pause at any time during the analysis, press <F8>. The autosampler will stop after the current sample and wait until the <F8> key is pressed

again. If the user presses the <F10> key, then the macro will stop immediately. The only way to resume a stopped analysis is to go to step 14.16 and restart the MERCURY macro.

15.30 When the autosampler has completed the analysis of ≤10 samples, the instrument will automatically analyze the CCB. The amount found in the CCB must not be greater than the absolute value of the reporting limit. If at any point in the decision tree below the CCB is within the limits, the macro will jump to step 14.31.

15.30.1 If the CCB does not meet the criteria, the macro will ask the user to place a new vial of the blank in autosampler position C1. Press and key to continue.

15.30.2 The instrument will then analyze the CCB again. If the CCB still does not meet the criteria, corrective actions will be taken. If a high level sample is carrying over, blanks will be analyzed until the system is clean. If there is no apparent cause for the out of control CCB, the analyst will allow the macro to autozero the instrument, and the CCB will be re-analyzed. If an autozero is required, all samples analyzed since the last acceptable CCB will be re-analyzed.

15.30.3 Please note that there is a possibility of an infinite loop occurring at this step. If the CCB still does not work after the instrument has been autozeroed, stop the macro and contact the metals lab supervisor for assistance. An acceptable CCB is required for sample data to be acceptable.

15.31 When the CCB reads back within limits, the CCV will automatically be analyzed. The CCV is the midpoint calibration standard analyzed as a sample with the concentration being read from the analytical curve. The percent recovery for the CCV must be within the control limits of 80%-120% of the true value. If the CCV recovers within the control windows, then the macro will jump to Step 14.32.

15.31.1 If the CCV does not fall within established control limits, the macro will ask the user to place another vial of the CCV in autosampler position C2. Press <Enter> to continue.

15.31.2 If the CCV still does not meet the criteria, then the macro will ask the user to prepare a new CCV and to place the vial for the new CCV in autosampler position C3. Press any key to continue.

15.31.3 If the CCV still does not work, stop the macro and contact the metals lab supervisor for assistance. An acceptable CCV is required for sample data to be acceptable.

15.32 If the CCV recovers within control limits, then start again at Step 14.29 with the next set of samples to be analyzed. If there are no more samples to analyze, then go to Step 14.33.

15.33 Check over the raw data to make sure that all of the samples are within the calibration range of the instrument. If any samples are above the calibration range of the curve, mark the sample number and appropriate dilution factor. Dilutions will need to be ran with the next batch.

16.0 CALCULATIONS AND DATA HANDLING

16.1 The concentration of each sample is read directly from the computer printout. Dilution factors should be taken into account in the ID/WT file.

16.1.1 Assuming that the instrument did not correct a concentration due to analyst error, the following calculation would be used:

$$C_{Final} = C_{Instr} * DF_{Digest} * DF_{Subseq}$$

where

C_{Final} is the final reported concentration of the analyte
C_{Instr} is the concentration as read from the instrument data printout
DF_{Digest} is the digestion dilution factor
DF_{Subseq} is any dilution done subsequent to the digestion dilution

16.2 All samples should be reported to the correct number of significant figures. The significant figure truncation should not be performed until all data calculation have taken place.

16.2.1 Solid samples must be reported in mg/kg.

16.2.1.1 For sample concentrations <100 mg/kg, report 2 significant figures.

16.2.1.2 For sample concentrations ≥100 mg/kg, report 3 significant figures.

16.2.1.3 For QC, always report one additional significant figure.

16.2.2 Aqueous samples must be reported in ug/L.

16.2.2.1 For sample concentrations <1 ppb, report 1 significant figure.

16.2.2.2 For sample concentrations ≥1 ppb and <100 ppb, report 2 significant figures.

16.2.2.3 For sample concentrations ≥100 ppb, report 3 significant figures.

16.2.2.4 For QC, always report one additional significant figure.

16.2.3 Extracted samples for all metals must be reported in mg/L.

16.2.3.1 For sample concentrations <0.001 mg/L, report 1 significant figure.

16.2.3.2 For sample concentrations ≥0.001 mg/L and <0.100 mg/L, report 2 significant figures.

16.2.3.3 For sample concentrations ≥0.100 mg/L, report 3 significant figures.

16.2.3.4 For QC, always report one additional significant figure.

17.0 DATA REPORTING AND DELIVERABLES

17.1 See appendices F, H, I, and J for data reporting.

18.0 QUALITY ASSURANCE

18.1 All quality control data should be maintained and available for easy reference or inspection.

18.2 A calibration curve must be prepared each day with a minimum of a reagent blank and three standards. The correlation coefficient must be ≥ 0.995. If this fails the problem must be corrected.

18.3 IDL and MDL studies must be performed on an annual basis.

18.3.1 To calculate an IDL, analyze ten cups of Blank solution in series. Calculate the standard deviation of a small population, and multiply this result by three. There is also a spreadsheet located on the computer network that will perform this calculation for you. Please see the metals lab supervisor if you need assistance in using this program.

18.3.2 To calculate an MDL, analyze ten cups of a low concentration standard, typically at a concentration at or near the detection limit. Calculate the standard deviation of a small population and multiply the result by the Student's T value for (n-1) degrees of freedom. If the concentration of the standard ran is greater than ten times the calculated MDL, and the concentration of the standard ran is above the detection limit reported to clients, the MDL must be performed at lower concentration. If the standard concentration is less than ten times the calculated MDL or if the standard used for the analysis was at or below the lowest reporting limit on LIMS, the MDL is acceptable. There is also a spreadsheet located on the computer network that will perform this calculation for you. Please see the metals lab supervisor if you need assistance in using this program.

18.4 Dilute, reprep, and reanalyze all samples that have concentrations greater than the highest standard of the analytical curve.

18.5 Include a minimum of one laboratory blank per sample digestion batch to determine if contamination or any memory effects are occurring. This laboratory blank must be carried through the sample preparation procedure.

18.6 Analyze one spike and one matrix spike duplicate (MSD) at a frequency of at least 1 in 20. A spike and matrix spike duplicate are samples into which a calculated extra amount of metal is pipetted in. Currently, the spike concentration is at 2.5 µg/L. The spike measures the accuracy of the sample preparation method and the MSD checks the precision of the method.

18.7 **Standard addition:** The Method of Standard Additions (MSA) shall be used for all EP extracts. MSA must also be performed on all TCLP extracts that have spike recoveries less than 50%. The standard-addition technique involves adding known amounts of standard to one or more aliquots of the process sample solution. This technique compensates for a sample constitute that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct additive interferences that cause a baseline shift. The simple version of this technique is the single addition method, in which identical aliquots of the sample solution, each of volume V_X, are taken. To the first (labeled A) is added a small volume V_S of a standard solution of concentration C_S. To the second (labeled B) is added the same volume V_S of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration C_X is calculated:

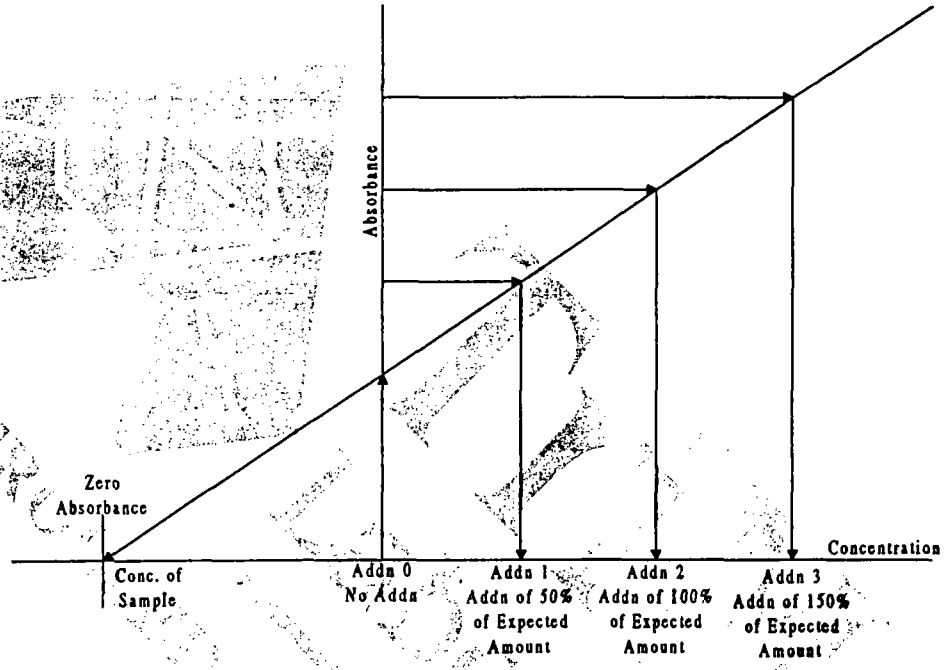
$$C_X = \frac{S_B * V_S * C_S}{(S_A - S_B) * V_X}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_S and C_S should be chosen so that S_A is roughly twice S_B on the average. It is best if V_S is made much less than V_X, and thus C_S is much greater than C_X, to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure. For the results of this technique to be valid, the following limitations must be taken into consideration:

1. The analytical curve must be linear and the correlation coefficient must be ≥ 0.995.
2. The chemical form of the analyte added must respond the same way as the analyte in the sample.
3. The interference effect must be constant over the working range of concern.
4. The signal must be corrected for any additive interference.

The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 2.

Figure 2 An Example of an MSA curve



NOTE: A method of standard addition must be performed on any TCLP sample whose spike recovery is not greater than 50%.

18.8 Check the instrument standardization by analyzing appropriate quality control check standards as follows.

NOTE: Steps 18.8.2 through 18.8.6 must be performed every time the instrument is calibrated.

18.8.1 Calibrate the instrument using at least three standards and a blank.

18.8.2 An ICB (Initial Calibration Blank) is analyzed after the calibration. The results of this blank are to be within ± 0.2 µg/L. If the blank is not within ± 0.2 µg/L, correct the problem, and restart the analysis from the beginning.

18.8.3 An ICV (Initial Calibration Verification) is placed after the ICB. Percent recovery limits are 80% to 120% of the concentration. If the percent recovery for this standard is not within the acceptance limits, correct the problem, re-calibrate if necessary, and restart the analysis.

18.8.4 An LCS (Laboratory Check Standard), which must come from a different primary source than the calibration standards, is placed after the ICV in each run. At the time of this revision, the diluted LCS was at a concentration of 3.0 µg/L. Percent recovery limits are 80% to 120% of the concentration unless control limits have been established. If the percent recovery for this standard is not within the acceptance limits, correct the problem. If the instrument had to be re-calibrated, restart the analysis from the beginning.

18.8.5 Verify the calibration after every 10 samples.

18.8.5.1 A CCV (Continuing Calibration Verification) is analyzed after every 10 samples, or sooner. Percent recovery limits are 80% to 120% of the concentration. If the percent recovery for this standard is not within the acceptance limits, correct the problem. When the problem has been corrected, analyze a CCV. If the CCV is within control limits, restart the analysis from the last good ICV or CCV. If the CCV is not within the control limits, repeat steps 18.8.1 through 18.8.4, and then reanalyze all samples since the last good ICV or CCV.

18.8.5.2 A CCB (Continuing Calibration Blank) is analyzed after the CCV. The results of this blank are to be within ± 0.2 µg/L. If the blank is not within ± 0.2 µg/L, correct the problem, and restart the analysis from the last good CCB or ICB.

18.8.6 At the end of each run, verify the calibration.

18.8.6.1 A CCV (Continuing Calibration Verification) is analyzed after every 10 samples, or sooner. Percent recovery limits are 80% to 120% of the concentration. If the percent recovery for this standard is not within the acceptance limits, correct the problem. When the problem has been corrected, analyze a CCV. If the CCV is within control limits, restart the analysis from the last good ICV or CCV. If the CCV is not within the control limits, repeat steps 18.8.1 through 18.8.4, and then reanalyze all samples since the last good ICV or CCV.

18.8.6.2 A CCB (Continuing Calibration Blank) is analyzed after the CCV. The results of this blank are to be within ± 0.2 µg/L. If the blank is not within ± 0.2 µg/L, correct the problem, and restart the analysis from the last good CCB or ICB.

18.9 A new analytical curve must be ran every 24 hours.

18.10 If the % RSD of the replicates for a sample is greater than 20% and the concentration of the sample is greater than the reporting limit, reanalyze the sample once. If the % RSD is again greater than 20% on a sample, qualify the data with data Qualifier number 10 "Duplicate injection precision not met for this analysis. The corresponding sample result must be considered estimated".

19.0 ANALYST CERTIFICATION/METHOD VALIDATION

19.1 Before the analysis of any actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running a one time analyst certification. While the analyst certification is not instrument dependent, this certification is required on every instrument that will be running samples to demonstrate instrument ability to generate acceptable accuracy and precision.

19.2 Prepare a quality control check sample spiking standard at a level that will give concentrations 1 mg/L.

19.3 Analyze the four distilled check samples following the SOP.

19.4 Calculate the average recovery (x) in mg/L, and the standard deviation of the recovery (s) in mg/L, for each analyte using the four results.

19.4.1 For each analyte x must be in the range 70-130% and s must be less than or equal to 20. If s and x for all analytes meet the acceptance criteria, the analyst certification is good. The analyst and the system are now authorized to run samples by this method. If any individual s exceeds the precision limit or any individual falls outside the range for accuracy, then the system performance is unacceptable for that analyte.

19.4.2 When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst must proceed according to 19.5.

19.5 Locate and correct the source of the problem and repeat the test for all analytes that failed to meet the criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds beginning with Section 19.1. Samples may not be analyzed by any analyst or on any instrument until the analyst certification has been successfully completed. Copies of the successful analyst certifications/method validations spreadsheet and raw data should be given to the Quality Assurance Manager.

20.0 REFERENCES

20.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 1, September, 1994, Method 7470A, "Mercury in Liquid Waste (Manual Cold-Vapor Technique)".

20.2 *Methods for the Determination of Metals in Environmental Samples,* Supplement I, May 1994, Revision 5.4, EMMC Version, "Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry", Method 245.1, Revision 3.0, May, 1994.

20.3 Gaskill, A, Compilation and Evaluation of RCRA Method Performance Data Work Assignment #2, EPA Contract No. 68-01-7075, September 1986.

20.4 Leeman Labs PS Series ICP/Eschelle Spectrometers Reference Manual Rev. 1.0, May 1990.

21.0 ATTACHMENTS/APPENDICES

See attached pages.

Approved By: _____ 12/4/98 (QA Manager) Approved By: _____ 12/4/98 (Area Manager)

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE

USEPA METHOD 5035

APPROVALS:

Organic Manager: _____ Date: 4/30/98
Jeff P. Glaser

QA Manager: _____ Date: 4/30/98
Rick D. Wilburn

Laboratory Manager: _____ Date: 4/30/98
Douglas E. Kriscunas

Procedure Number: GR-04-105
Revision Number: 0.0

Date Initiated: 4/30/98
Effective Date: 4/30/98

Date Revised: New
Pages Revised: New

By: Jeff Glaser

Total Number of Pages: 25

1.0 SCOPE AND APPLICATION

1.1 This method describes a closed-system purge-and-trap process for the analysis of volatile organic compounds (VOCs) in solid materials (e.g., soils, sediments, and solid waste). While the method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs and for oily wastes. For these high concentration and oily materials, sample collection and preparation are performed using the procedures described here, and sample introduction is performed using the aqueous purge-and-trap procedure in Method 5030B. These procedures may be used in conjunction with any appropriate determinative gas chromatographic procedure, including, but not limited to, Methods 8015, 8021B, and 8260B.

1.2 Procedures are included for preparing high concentration samples (samples containing VOC levels of >1 mg/kg) for purging by Method 5030B. Procedures are also included for addressing oily wastes that are soluble in a water-miscible solvent (also purged using Method 5030B). The closed-system purge-and-trap equipment employed for low concentration samples is NOT appropriate for soil samples preserved in the field with methanol. Such samples should be analyzed using Method 5030B.

1.3 Method 5035 can be used for most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile, water-soluble compounds can be included in this analytical technique. However, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency.

1.4 Method 5035, in conjunction with Method 8015 (GC/FID), may be used for the analysis of the aliphatic hydrocarbon fraction in the light ends of total petroleum hydrocarbons, e.g., gasoline. For the aromatic fraction (BTEX), use Method 5035 and Method 8021B (GC/PID). A total determinative analysis of gasoline fractions may be obtained using Method 8021B in series with Method 8015.

1.5 As with any preparative method for volatiles, samples should be screened to avoid contamination of the purge-and-trap system by samples that contain very high concentrations of purgeable material above the calibration range of the low concentration method. In addition, because the sealed sample container cannot be opened to remove a sample aliquot without compromising the integrity of the sample, multiple sample aliquots should be collected to allow for screening and reanalysis.

1.6 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Update III, Revision 2, December 1996, Method 5035.

3.0 SUMMARY OF PROCEDURE

3.1 Low concentration soil method - generally applicable to soils and other solid samples with VOC concentrations in the range of 0.0005 to 1 mg/kg. Volatile organic compounds (VOCs) are determined by collecting approximately a 5 g sample, weighed in the field at the time of collection, and placing it in a pre-weighted 40 ml VOA vial with a septum-sealed screw-cap that already contains a stirring bar and a sodium bisulfate preservative solution. The vial is sealed and shipped to the laboratory or appropriate analysis site. The entire vial is then placed, unopened, onto the autosampler. Immediately before analysis, organic-free reagent water, surrogates, and internal standards (if applicable) are automatically added without opening the sample vial. The vial containing the sample is heated to 40°C, the sample is magnetically stirred, and the volatiles are purged onto an appropriate trap using helium gas. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped sample components into a gas chromatograph for analysis by an appropriate determinative method.

3.2 High concentration soil method - generally applicable to soils and other solid samples with VOC concentrations greater than 1 mg/kg. The sample introduction technique described above is not applicable to all samples, particularly those containing high concentrations of VOCs which may overload either the volatile trapping material or exceed the working range of the determinative instrument system (e.g., GC/MS, GC/FID, GC/PID, etc.). In such instances, this method describes two sample collection options and the corresponding sample purging procedures.

3.2.1 The first option is to collect a bulk sample in a 60 or 125 ml glass container without the use of the sodium bisulfate preservative solution. A 10 gram portion of that sample is removed from the container in the laboratory and is added to 10 ml of methanol to dissolve the volatile organic constituents. An aliquot of the solution is added to 5 ml of reagent water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, purged using Method 5030B, and analyzed by an appropriate determinative method. This is the least desirable option as the procedure involves opening the vial and removing a portion of the soil, which will cause the loss of some of the volatile constituents.

3.2.2 The second option is to collect approximately a 25 g sample in a pre-weighed vial with a septum-sealed screw-cap that contains 25 ml of methanol. At the time of analysis, surrogates are added to the vial, then an aliquot of the solvent is removed from the vial, purged using Method 5030B, and analyzed by an appropriate determinative method.

3.3 High concentration oily waste method - generally applicable to oily samples with VOC concentrations greater than 0.20 mg/kg that can be diluted in a water-miscible solvent (samples that contain oily materials that are not soluble in water-miscible solvents must be prepared according to Method 3585). After demonstrating that a test aliquot of the sample is soluble in methanol or polyethylene glycol (PEG), a separate aliquot of the sample is spiked with surrogates and diluted in the appropriate solvent. An aliquot of the solution is added to 5 ml of reagent water in a purge tube, taking care to ensure that a floating layer of oil is not present in the purge tube. Internal standards (if applicable) are added to the solution which is then purged using Method 5030B and analyzed by an appropriate determinative method.

4.0 PARAMETER OR COMPOUND LIST

4.1 See determinative method for parameter list.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP number GR-03-124, *Volatile Organic Compounds By Purge And Trap Capillary Column Gas Chromatography/Mass Spectrometry*, most current version.

5.2 TriMatrix SOP number GR-03-105, Volatile Organic Compounds By Purge And Trap Capillary Column Gas Chromatography With Photoionization And Electrolytic Conductivity Detectors in Series, most current version.

5.3 TriMatrix SOP number GR-03-121, *Method for The Determination of Gasoline Range Organics*, most current version.

5.4 TriMatrix SOP number GR-03-124, *Volatile Organic Laboratory Corrective Actions*, most current version.

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Whether purged or injected directly on column, interferences naturally present in the samples can cause elevated detection limits and interfere with the analysis. Naturally occurring interferences can vary considerably from site to site.

6.2 Sample contamination can also cause raised detection limits or false positives, and can come from a variety of sources. Improper sampling techniques can contaminate the sample at the job site. During shipment and storage, volatile organics (particularly methylene chloride and fluorocarbons) can diffuse through the septum and into the sample. A trip blank prepared from reagent water and carried through the sampling and handling protocol will serve as a check on such contamination.

6.3 During analysis, contamination can come from impurities in the purge gas, or from organic compounds outgassing from the plumbing ahead of the trap where they were deposited by a previously analyzed high level sample. To reduce this, the use of non-Teflon plastic tubing, non-Teflon thread sealants, and flow controllers with rubber components in the purging device have been eliminated where possible.

6.4 Contamination by carryover can occur whenever high level and low level samples are sequentially analyzed. To reduce carryover, the purging device is rinsed with reagent water between samples. When practical, samples with unusually high concentrations of analytes should be followed by an analysis of organic-free reagent water to check for cross-contamination. If compounds present in high concentrations are also present in subsequent samples, the analyst must demonstrate that the compounds are not due to

carryover. However, if the compounds are not present in subsequent samples, then reanalysis is not necessary.

6.5 The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required. If the contamination is persistent, the complete purge and trap system should be purged first with 100°C reagent water, then, if necessary, with methanol. If methanol is used, either disconnect the trap or install a blank trap, as the methanol will adversely affect the trap's performance.

6.6 For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high organohalide levels, it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105°C oven between analyses.

6.7 The laboratory where volatile analysis is performed should be completely free of solvents. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride and other solvents, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination.

6.8 For corrective actions, see TriMatrix SOP number GR-03-124, *Volatile Organic Laboratory Corrective Actions*, most current version.

7.0 SAFETY PRECAUTIONS

7.1 The analyst must comply with all standard operating procedures for health and safety as outlined in the TriMatrix Laboratories, Inc. Laboratory Safety Manual.

7.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets should also be made available to all personnel involved in the chemical analysis.

7.3 The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichloroethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromomethane, tetrachloroethene, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Preparation of Sample Vials

8.1.1 The specific sample containers required will depend on the purge-and-trap system to be employed. TriMatrix Laboratories currently employs the needle sparger type purging device for all low level soils. These systems utilize standard 40-ml borosilicate glass VOA vials. Some systems employ 40-ml clear vials with a special frit and equipped with two PTFE-faced silicone septa. Consult the purge-and-trap system manufacturer's instructions regarding the suitable specific vials, septa, caps, and mechanical agitation devices.

8.1.2 The specific preparation procedures for sample vials depend on the expected concentration range of the sample, with separate preparation procedures for low concentration soil samples and high concentration soil and solid waste samples. Sample vials should be prepared in a laboratory or other controlled environment, sealed, and shipped to the field location. Gloves should be worn during the preparation steps.

8.1.3 Low concentration soil samples: The following steps apply to the preparation of vials used in the collection of low concentration soil samples to be analyzed by the closed-system purge-and-trap equipment described in Method 5035.

8.1.3.1 Add a clean magnetic stirring bar to each clean vial.

8.1.3.2 Add approximately 1 g of sodium bisulfate preservative to each vial. If samples markedly smaller or larger than 5 g are to be collected, adjust the amount of preservative added to correspond to approximately 0.2 g of preservative for each 1 g of sample. Enough sodium bisulfate should be present to ensure a sample pH of ≤2.

8.1.3.3 Add 5 ml of organic-free reagent water to each vial. The water and the preservative will form an acid solution that will reduce or eliminate the majority of the biological activity in the sample, thereby preventing biodegradation of the volatile target analytes.

8.1.3.4 Seal the vial with the screw-cap and septum seal. If the double-ended, fritted vials are used, seal both ends as recommended by the manufacturer.

8.1.3.5 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label.

8.1.3.6 Weigh the prepared vial to the nearest 0.01 g, record the tare weight, and write it on the label.

8.1.3.7 Because volatile organics will partition into the headspace of the vial from the aqueous solution and will be lost when the vial is opened, surrogates, matrix spikes, and internal standards (if applicable) should only be added to the vials after the sample has been added to the vial. These standards should be introduced back in the laboratory, either manually by puncturing the septum with a small gauge needle or automatically by the sample introduction system, just prior to analysis.

8.1.4 High concentration soil samples collected without a preservative: When high concentration samples are collected without a preservative, a variety of sample containers may be employed, including 60-ml glass vials with septum seals. This collection technique should only be used when the solubility of the sample in methanol is questionable.

8.1.5 High concentration soil samples collected and preserved in the field: The following steps apply to the preparation of vials used in the collection of high concentration soil samples to be preserved in the field with methanol and analyzed by the aqueous purge-and-trap equipment described in Method 5030B.

8.1.5.1 60 or 125 ml jars with screw cap lids and teflon liners are purchased pre-tared or tared by TriMatrix. A label is affixed to the jar prior to weighing, and the weight is taken with the lid and liner in place. The weight of the jar is recorded on the label (the weight of the ink is negligible) to the nearest 0.01 g.

8.1.5.2 Purchased 25 ml ampules of purge and trap methanol are included for each sample container. If the samples are insoluble in methanol but are soluble in PEG, PEG will be provided in place of the methanol.

8.1.6 Oily waste samples: When oily waste samples are known to be soluble in methanol or PEG, sample vials may be prepared as described in Sec. 8.1.5, using the appropriate solvent. However, when the solubility of the waste is unknown, the sample should be collected without the use of a preservative, in a vial such as that described in Sec. 8.1.4.

8.2 Sample Collection

8.2.1 Collect the sample according to the procedures outlined in the sampling plan. As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Several techniques may be used to transfer a sample to the relatively narrow opening of the low concentration soil vial. These include devices such as the EnCore™ sampler, the Purge-and-Trap Soil Sampler™, and a cut plastic syringe. Always wear gloves whenever handling the tared sample vials.

8.2.2 Low Concentration Soil Samples

8.2.2.1 Using an appropriate sample collection device, collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

8.2.2.2 Using the sample collection device, add about 5 g (2 - 3 cm) of soil to the sample vial containing the preservative solution. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Store samples on ice at 4°C.

NOTE: Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution in the low concentration sample vial. If the amount of gas generated is very small (i.e., several ml), any loss of volatiles as a result of such effervescence may be minimal if the vial is sealed quickly. However, if larger amounts of gas are generated, not only may the sample lose a significant amount of analyte, but the gas pressure may shatter the vial when the sample vial is sealed. *Therefore, when samples are known or suspected to contain high levels of* carbonates, a test sample should be collected, added to a vial, and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and collect low concentration samples in vials that do not contain the preservative solution. This must be noted on the chain-of-custody.

8.2.2.3 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5 g of sample were added. The balance should be calibrated in the field using an appropriate Class S weight for the sample containers employed. Record the weight of the sealed vial containing the sample to the nearest 0.01 g.

8.2.2.4 Alternatively, collect several trial samples with plastic syringes. Weigh each trial sample and note the length of the soil column in the syringe. Use this data to determine the length of soil in the syringe that corresponds to 5.0 ± 0.5 g. Discard each trial sample.

8.2.2.5 As with the collection of aqueous samples for volatiles, collect at least two replicate samples. This will allow the laboratory an additional sample for reanalysis. The second sample should be taken from the same soil stratum or the same section of the solid waste being sampled, and within close proximity to the location from which the original sample was collected.

8.2.2.6 In addition, since the soil vial cannot be opened without compromising the integrity of the sample, at least one additional aliquot of sample must be collected for screening, dry weight determination, and high concentration analysis (if

necessary). This third aliquot may be collected in a 60-ml glass vial or a third 40-ml soil sample vial. However, this third vial *must not* contain the sample preservative solution, as an aliquot will be used to determine dry weight.

8.2.2.7 If samples are known or expected to contain target analytes over a wide range of concentrations, thereby requiring the analyses of multiple sample aliquots, it may be advisable and practical to take an additional sample aliquot in a low concentration soil vial containing the preservative, but collecting only 1-2 g instead of the 5 g collected in Secs. 8.2.2.1 and 8.2.2.2. This aliquot may be used for those analytes that exceed the instrument calibration range in the 5 g analysis.

8.2.2.8 The EnCore™ sampler has not been thoroughly evaluated by EPA as a sample storage device. While preliminary results indicate that storage in the EnCore™ device may be appropriate for up to 48 hours, samples collected in this device should be transferred to soil sample vials as soon as possible, or analyzed within 48 hours.

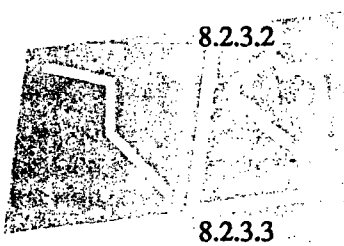
8.2.2.9 The collection of low concentration soil samples in vials that contain methanol is not appropriate for samples analyzed with the closed-system purge-and-trap equipment described in this method.

8.2.3 High concentration soil samples preserved in the field: The collection of soil samples in vials that contain methanol has been suggested by some as a combined preservation and extraction procedure. However, this procedure is not appropriate for use with the low concentration soil procedure described in this method.

NOTE: The use of methanol preservation has not been formally evaluated by EPA and analysts must be aware of two potential problems. First, the use of methanol as a preservative and extraction solvent introduces a significant dilution factor that will raise the method quantitation limit beyond the operating range of the low concentration direct purge-and-trap procedure (0.0005-1.0 mg/kg). The exact dilution factor will depend on the masses of solvent and sample, but generally exceeds 50, and may make it difficult to demonstrate compliance with regulatory limits or action levels for some analytes. Because the analytes of interest are volatile, the methanol extract cannot be concentrated to overcome the dilution problem. Thus, for samples of unknown composition, it may still be necessary to collect an aliquot for analysis by this closed-system procedure and another aliquot preserved in methanol and analyzed by other procedures. The second problem is that the addition of methanol to the sample is likely to cause the sample to fail the ignitability characteristic, thereby making the unused sample volume a hazardous waste.

8.2.3.1 When samples are known to contain volatiles at concentrations high enough that the dilution factor will not preclude obtaining results within the calibration range

of the appropriate determinative method, a sample may be collected and immediately placed in a sample vial containing purge-and-trap grade methanol.



8.2.3.2 Using an appropriate sample collection device, collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

8.2.3.3 Using the sample collection device, add about 25 g (4 - 6 cm) of soil to the sample vial containing 25 ml of methanol. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Store samples on ice at 4°C.

8.2.3.4 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 25.0 ± 0.5 g of sample were added. The balance should be calibrated in the field using an appropriate Class S weight for the sample containers employed. Record the weight of the sealed vial containing the sample to the nearest 0.01 g.

8.2.3.5 Alternatively, collect several trial samples with plastic syringes. Weigh each trial sample and note the length of the soil column in the syringe. Use these data to determine the length of soil in the syringe that corresponds to 25.0 ± 0.5 g. Discard each trial sample.

8.2.3.6 Other sample weights and volumes of methanol may be employed, provided that the analyst can demonstrate that the sensitivity of the overall analytical procedure is appropriate for the intended application.

8.2.3.7 The collection of at least one additional sample aliquot is required for the determination of the dry weight, as described in Sec. 8.2.2.6. Samples collected in methanol should be shipped as described in Sec. 8.4.1, and must be clearly labeled as containing methanol, so that the samples are not analyzed using the closed-system purge-and-trap equipment described in this procedure.

8.2.4 High concentration soil sample not preserved in the field: The collection of high concentration soil samples that are not preserved in the field generally follows similar procedures as for the other types of samples described in Secs. 8.2.2 and 8.2.3, with the obvious exception that the sample vials contain neither the aqueous preservative solution nor methanol. However, when field preservation is not employed, it is better to collect a larger volume sample, filling the sample container as full as practical in order to minimize headspace. Such collection procedures generally do not require the collection of a separate aliquot for dry weight determination, but it may be advisable to collect a second sample aliquot for screening

purposes, in order to minimize the loss of volatiles in either aliquot. This collection technique should only be used when the solubility of the sample in methanol is questionable.

8.2.5 Oily waste samples: The collection procedures for oily samples depend on knowledge of the waste and its solubility in methanol or other solvents.

8.2.5.1 When an oily waste is known to be soluble in methanol or PEG, the sample may be collected in a vial containing such a solvent (see Sec. 8.1.6), using procedures similar to those described in Sec. 8.2.3.

8.2.5.2 When the solubility of the oily waste is not known, the sample should either be collected in a vial without a preservative, as described in Sec. 8.2.4, or the solubility of a trial sample should be tested in the field, using a vial containing solvent. If the trial sample is soluble in the solvent, then collect the oily waste sample as described in Sec. 8.2.3. Otherwise, collect an unpreserved sample as described in Sec. 8.2.4.

8.3 Trip Blanks

8.3.1 Trip blanks should accompany each sample set into the field and back. A sample set is composed of the samples collected from the same general sample site at approximately the same time. At the laboratory, fill trip blank sample bottles with reagent water, seal, and ship to the sampling site along with empty sample bottles. Wherever a set of samples is shipped and stored, it is accompanied by the trip blanks.

8.3.2 Follow Sec. 8.1.3 and 8.1.5 to add preservative to trip blanks.

8.4 Sample Handling and Shipment

8.4.1 All samples for volatiles analysis should be cooled by approximately 4°C, packed in appropriate containers, and shipped to the laboratory on ice, as described in the sampling plan.

8.5 Sample Storage

8.5.1 Once in the laboratory, store samples at 4°C until analysis. The sample storage area should be free of organic solvent vapors.

8.5.2 All samples should be analyzed as soon as practical, and within the designated holding time from collection. Samples not analyzed within the designated holding time must be noted, the data estimated, and considered minimum values.

8.5.3 When the low concentration samples are strongly alkaline or highly calcareous in nature, the sodium bisulfate preservative solution may not be strong enough to reduce the pH of the

soil/water solution to below 2. Therefore, when low concentration soils to be sampled are known or suspected to be strongly alkaline or highly calcareous, additional steps may be required to preserve the samples. Such steps include: addition of larger amounts of the sodium bisulfate preservative to non-calcareous samples, storage of low concentration samples at -10°C (taking care not to fill the vials so full that the expansion of the water in the vial breaks the vial), or significantly reducing the maximum holding time for low concentration soil samples. Whichever steps are employed, they should be clearly described in the sampling and QA project plans and distributed to both the field and laboratory personnel. See Sec. 8.2.2.2 for additional information.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Sample Containers: Either 40-ml clear vials with a special frit and two PTFE-faced silicon septa screw-caps, or regular 40-ml clear vials with PTFE-faced silicon septa screw-caps.(See Sec. 8.1.1)

9.2 Purge-and-Trap System: consisting of a unit that automatically adds water, surrogates, and internal standards (if applicable) to a vial containing the sample, purges the VOCs using an inert gas stream while agitating the contents of the vial, and also traps the released VOCs for subsequent desorption into the gas chromatograph. Such systems are commercially available from several sources and shall meet the following specifications. The following descriptions are for informative purposes only. The specific apparatus and conditions used are specified in the determinative SOPs.

9.2.1 The purging device should be capable of accepting a vial sufficiently large to contain a 5 g soil sample plus a magnetic stirring bar and 10 ml of water. The device must be capable of heating a soil vial to at least 40°C and holding it at that temperature while the inert purge gas is allowed to pass through the sample. The device should also be capable of introducing at least 5 ml of organic-free reagent water along with surrogate and internal standards into the sample vial while trapping the displaced headspace vapors. It must also be capable of agitating the sealed sample during purging, (e.g., using a magnetic stirring bar added to the vial prior to sample collection). The analytes being purged must be quantitatively transferred to an absorber trap. The trap must be capable of transferring the absorbed VOCs to the gas chromatograph (see Sec. 9.2.2).

9.2.2 A variety of traps and trapping materials may be employed with this method. The choice of trapping material depends on the analytes of interest. Whatever trap is employed, it must demonstrate sufficient adsorption and desorption characteristics to meet the quantitation limits of all the target analytes for a given project and the QC requirements in Method 8000 and the determinative method. The most difficult analytes are generally the gases, especially dichlorodifluoromethane. The trap must also be capable of desorbing the late eluting target analytes.

9.2.2.1 The trap used to develop this method was 25 cm long, with an inside diameter of 0.105 inches, packed with Carbopack/Carbosieve (Supelco, Inc.).

9.2.2.2 The standard trap used in other EPA purge-and-trap methods is also acceptable. That trap is 25 cm long and has an inside diameter of at least 0.105 inches. Starting from the inlet, the trap contains the equal amounts of the adsorbents listed below. It is recommended that 1.0 cm of methyl silicone-coated packing (35/60 mesh, Davison, grade 15 or equivalent) be inserted at the inlet to extend the life of the trap. If the analysis of dichlorodifluoromethane or other fluorocarbons of similar volatility is not required, then the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35°C are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap.

9.2.2.2.1 2,6-Diphenylene oxide polymer - 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

9.2.2.2.2 Methyl silicone packing - OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

9.2.2.2.3 Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26, or equivalent, by crushing through 26 mesh screen.

9.2.2.3 Trapping materials other than those listed above also may be employed, provided that they meet the specifications in Sec. 9.2.3, below.

NOTE: Check the responses of the brominated compounds when using alternative charcoal traps (especially Vocarb 4000), as some degradation has been noted when higher desorption temperatures (especially above 240 - 250°C) are employed. 2-Chloroethyl vinyl ether is degraded on a Vocarb 4000 trap, but performs adequately when a Vocarb 3000 trap is used. The primary criterion, as stated above, is that all target analytes meet the sensitivity requirements for a given project.

9.2.3 The desorber for the trap must be capable of rapidly heating the trap to the temperature recommended by the trap material manufacturer, prior to the beginning of the flow of desorption gas. Several commercial desorbers (purge-and-trap units) are available.

9.2.4 Purge and Traps to be utilized by TriMatrix Laboratories with this method are listed below:

9.2.4.1 Dynatech PTA 30 W/S Autosampler/OI Analytical Model 4560 Concentrator

9.2.4.2 Dynatech Dynatrap Purge and Trap Autosampler/Concentrator

9.2.4.3 Dynatech PTA 30 W/S Autosampler/Tekmar Model LSC 2000 Concentrator

9.2.4.4 O.I. Analytical 4552 Autosampler/Tekmar Model LSC 2000 Concentrator

9.3 Syringes and Syringe Valves

9.3.1 5 and 25-ml glass hypodermic syringes with Luer-Lok (or equivalent) tip

9.3.2 2-way syringe valves with Luer ends

9.3.3 25-ul micro syringe with a 2 inch x 0.006 inch ID, 22° bevel needle (Hamilton #702N or equivalent)

9.3.4 Micro syringes - 2.0, 10, 25, 50, and 100-ul

9.3.5 Syringes - 0.5, 1.0, and 5-ml, gas tight with shut-off valve

9.4 Miscellaneous

9.4.1 Glass vials

9.4.1.1 60-ml, septum-sealed, to collect samples for screening, dry weight determination

9.4.1.2 40-ml, screw-cap, PTFE-lined septum-sealed. Examine each vial prior to use to ensure that the vial has a flat, uniform sealing surface

9.4.2 Top-loading balance - capable of accurately weighing to 0.01 g

9.4.3 Glass scintillation vials - 20-ml, with screw-caps and PTFE liners, or glass culture tubes with screw-caps and PTFE liners, for dilution of oily waste samples

9.4.4 Volumetric flasks - Class A, 10-ml, 50-ml, and 100-ml, with ground glass stoppers

9.4.5 2-ml glass vials, for GC autosampler - used for oily waste samples extracted with methanol or PEG

9.4.6 Spatula, stainless steel - narrow enough to fit into a sample vial

9.4.7 Disposable Pasteur pipets

9.4.8 Magnetic stirring bars - PTFE- or glass-coated, of the appropriate size to fit the sample vials. Consult manufacturer's recommendation for specific stirring bars. Stirring bars may be reused, provided that they are thoroughly cleaned between uses. Consult the manufacturers of the purging device and the stir bars for suggested cleaning procedures.

9.5 Field Sampling Equipment

9.5.1 Purge-and-Trap Soil Sampler - model 3780PT (Associated Design and Manufacturing Company, 814 North Henry Street, Alexandria, VA 22314), or equivalent.

9.5.2 EnCore™ sampler - (En Chem, Inc., 1795 Industrial Drive, Green Bay, WI 54302), or equivalent.

9.5.3 Alternatively, disposable plastic syringes with a barrel smaller than the neck of the soil vial may be used to collect the sample. The syringe end of the barrel is cut off prior to sampling. One syringe is needed for each sample aliquot to be collected.

9.5.4 Portable balance - For field use, capable of weighing to 0.01g.

9.5.5 Balance weights - Balances employed in the field should be checked against an appropriate Class S reference weight at least once daily, prior to weighing any samples, or as described in the sampling plan. The specific weights used will depend on the total weight of the sample container, sample, stirring bar, reagent water added, cap, and septum.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Gas Chromatograph

10.1.1 Change septa weekly or as needed

10.1.2 Change gas scrubber traps as needed

10.1.3 Clip or replace capillary column as needed

10.1.4 Check column head pressure daily

10.1.5 Check cylinder gas tank pressure daily, and change tank if necessary

10.2 Detectors

10.2.1 Check detector signals daily

10.2.2 Clean or replace detector parts as needed (PID lamps, FID jets, MS source, etc.)

10.3 Purge and Trap System

10.3.1 Bake out trap daily prior to use

10.3.2 Check and record purge flow weekly

10.3.3 Check and fill surrogate and internal standard modules daily

10.3.4 Check concentrator pressure daily

10.3.5 Empty waste bottle and fill rinse bottle daily

11.0 CHEMICALS AND REAGENTS

11.1 Organic-free reagent water

11.2 Methanol, CH_3OH - purge-and-trap quality or equivalent. Store away from other solvents

11.3 Polyethylene glycol (PEG), $H(OCH_2CH_2)_nOH$ - free of interferences at the detection limit of the target analytes

11.4 Low concentration sample preservative

11.4.1 Sodium bisulfate, $NaHSO_4$ - ACS reagent grade or equivalent

11.4.2 The preservative should be added to the vial prior to shipment to the field, and must be present in the vial prior to adding the sample

11.5 See the determinative method for guidance on internal standards and surrogates to be employed in this procedure.

12.0 STANDARDS PREPARATION

12.1 See the determinative method for standards preparation procedures.

13.0 SAMPLE PREPARATION

13.1 Sample Screening

13.1.1 It is highly recommended that all samples be screened prior to the purge-and-trap GC or GC/MS analysis. Samples may contain higher than expected quantities of purgeable organics that will contaminate the purge-and-trap system, thereby requiring extensive cleanup and instrument maintenance. The screening data are used to determine which is the appropriate sample preparation procedure for the particular sample, the low concentration closed-system direct purge-and-trap method (Sec. 15.1), the high concentration (methanol extraction) method

(Sec. 15.2), or the nonaqueous liquid (oily waste) methanol or PEG dilution procedure (Sec. 15.3).

13.1.2 The analyst may employ any appropriate screening technique. Two suggested screening techniques employing SW-846 methods are:

13.1.2.1 Automated headspace (Method 5021) using a gas chromatograph (GC) equipped with a photoionization detector (PID) and an electrolytic conductivity detector (ELCD) in series.

13.1.2.2 Extraction of the sample with hexadecane (Method 3820), and analysis of the extract using a GC equipped with a FID and/or an ECD.

13.1.3 The analyst may inject a calibration standard containing the analytes of interest at a concentration equivalent to the upper limit of the calibration range of the low concentration soil method. The results from this standard may be used to determine when the screening results approach the upper limit of the low concentration soil method. There are no linearity or other performance criteria associated with the injection of such a standard, and other approaches may be employed to estimate sample concentrations.

13.1.4 Use the low concentration closed-system purge-and-trap method (Sec. 15.1) if the estimated concentration from the screening procedure falls within the calibration range of the selected determinative method. If the concentration exceeds the calibration range of the low concentration soil method, then use either the high concentration soil method (Sec. 15.2), or the oily waste method (Sec. 15.3).

14.0 CALIBRATION PROCEDURES

14.1 Initial Calibration for Low Concentration Method

14.1.1 Assemble a purge-and-trap device that meets the specifications in Sec. 9.2 and that is connected to a gas chromatograph or a gas chromatograph/mass spectrometer system.

14.1.2 Before initial use, a Carbopack/Carbosieve trap should be conditioned overnight at 245°C by backflushing with an inert gas flow of at least 20 ml/minute. If other trapping materials are substituted for the Carbopack/Carbosieve, follow the manufacturers recommendations for conditioning. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 minutes at 245°C with backflushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

14.1.3 If the standard trap in Sec. 9.2.2.2 is employed, prior to initial use, the trap should be conditioned overnight at 180°C by backflushing with an inert gas flow of at least 20

ml/minute, or according to manufacturer's recommendations. Vent the trap effluent to the hood, not the hood, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 minutes at 180°C with backflushing.

14.1.4 Establish the purge-and-trap instrument operating conditions. Adjust the instrument to inject 5 ml of water, and pre-heat the sample to 40°C for 1.5 minutes before commencing the purge process.

14.1.5 Prepare a minimum of five (5) initial calibration standards containing all the analytes of interest and surrogates, as described in the appropriate analytical method. The calibration standards are prepared in organic-free reagent water. **The volume of organic-free reagent water used for calibration must be the same volume used for sample analysis (normally 5 ml added to the vial before shipping it to the field plus the organic-free reagent water added by the instrument). The calibration standards should also contain approximately the same amount of the sodium bisulfate preservative as the sample (e.g., ~1 g), as the presence of the preservative will affect the purging efficiencies of the analytes. The internal standard solution must be added automatically, by the instrument, in the same fashion as used for samples.** Place the soil vial containing the solution in the instrument carousel. In order to calibrate the surrogates using standards at five (5) concentrations, it will be necessary to disable the automatic addition of surrogates to each vial containing a calibration standard. **Prior to purging, pre-heat the sample vial to 40°C for 1.5 minutes.**

14.1.6 Carry out the purge-and-trap procedure as outlined in Sec. 15.0

14.1.7 Calculate calibration factors (CF) or response factors (RF) for each analyte of interest using the procedures described in the appropriate method. Evaluate the linearity of the calibration data, or choose another calibration model, as described in the specific determinative method.

14.1.8 For GC/MS analysis, a system performance check must be made before this calibration curve is used (see Method 8260B). If the purge-and-trap procedure is used with Method 8021B, evaluate the response for the following four compounds: chloromethane, 1,1-dichloroethane, bromoform, and 1,1,2,2-tetrachloroethane. They are used to check for proper purge flow and to check for degradation caused by contaminated lines or active sites in the system.

14.1.8.1 Chloromethane is the most likely compound to be lost if the purge flow is too fast.

14.1.8.2 Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response.

14.1.8.3 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

14.1.9 When analyzing for very late eluting compounds with Method 8021B (i.e., hexachlorobutadiene, 1,2,3-trichlorobenzene, etc.), cross-contamination and memory effects from a high concentration sample or even the standard are a common problem. Extra rinsing of the purge chamber after analysis normally corrects this. The newer purge-and-trap systems often overcome this problem with better bakeout of the system following the purge-and-trap process. Also, the charcoal traps retain less moisture and decrease the problem.

14.1.10 **NOTE: THE ADDITION OF THE SODIUM BISULFATE PRESERVATIVE WILL DEGRADE THE PRESENCE OF 2-CHLOROETHYL VINYL ETHER IN BOTH STANDARDS AND SAMPLES. IT HAS BEEN DEMONSTRATED BY TRIMATRIX LABORATORIES THAT THIS COMPOUND IS UNABLE TO PRODUCE ACCEPTABLE RESULTS USING THIS METHOD.**

14.2 Calibration verification: Refer to the specific method for details on calibration verification. A single standard near the mid-point of calibration range is used for verification. **This standard should also contain approximately 1 g of sodium bisulfate.**

14.3 When analyzing samples by methods other than the closed-system low-concentration purge-and-trap method, calibration should be performed as indicated in the determinative methods.

15.0 ANALYTICAL PROCEDURE

15.1 Low concentration soil method (approximate concentration range of 0.0005 to 1 mg/kg - dependent upon the determinative method and the sensitivity of each analyte).

15.1.1 This method is designed for a 5 g sample size, but smaller sample sizes may be used. The soil vial is hermetically sealed at the sampling site, and MUST remain so in order to guarantee the integrity of the sample. Gloves must be worn when handling the sample vial since the vial has been tared. If any soil is noted on the exterior of the vial or cap, it must be carefully removed prior to weighing. **Weigh the vial and contents to the nearest 0.01 g, even if the sample weight was determined in the field, and record this weight.** This second weight provides a check on the field sampling procedures and provides additional assurance that the reported sample weight is accurate. **Data users should be advised on significant discrepancies between the field and laboratory weights.**

15.1.2 Remove the sample vial from storage and allow it to warm to room temperature. Shake the vial gently, to ensure that the contents move freely and that stirring will be effective. Place the sample vial in the instrument carousel according to the manufacturer's instructions.

15.1.3 Without disturbing the hermetic seal on the sample vial, add 7 ml of organic-free reagent water, the internal standards, and the surrogate compounds. This is carried out using the automated sampler. Other volumes of organic-free reagent water may be used, however, **it is imperative that all samples, blanks, and calibration standards have exactly the same final**

volume of organic-free reagent water. Prior to purging, heat the sample to 40°C for 1.5 minutes.

15.1.4 For the sample selected for matrix spiking, add the matrix spiking solution described in the appropriate method. The concentration of spiking solution and the amount added will vary by method and by instrument detector linearity.

15.1.5 Purge the sample with helium at a flow rate up to 45 ml/minute (the flow rate may vary from 30 to 50 ml/min., depending on the target analyte group) for 11 minutes while the sample is being agitated with the magnetic stirring bar. The purged analytes are allowed to flow out of the vial through a glass-lined transfer line to a trap packed with suitable sorbent materials.

15.1.6 Sample Desorption

15.1.6.1 After the 11 minute purge, place the purge-and-trap system in the desorb mode and preheat the trap to 245°C without a flow of desorption gas. Start the flow of desorption gas at 20 ml/minute for two minutes. Begin the temperature program of the gas chromatograph and start data acquisition.

15.1.7 After desorbing the sample, recondition the trap by baking out the purge-and-trap system at 245°C (or other temperature recommended by the manufacturer of the trap packing materials). After approximately 10 minutes, turn off the trap heater and halt the purge flow through the trap. When the trap is cool, analysis of the next sample can begin.

15.1.8 Perform qualitative and quantitative analysis following the guidance given in the determinative method. If the concentration of any target analyte exceeds the calibration range of the instrument, it will be necessary to reanalyze the sample by the high concentration method. Such reanalyses need only address those analytes for which the concentration exceeded the calibration range of the low concentration method. Alternatively, if a sample aliquot of 1-2 g was also collected (see Sec. 8.2.2.7), it may be practical to analyze that aliquot for the analytes that exceeded the instrument calibration range in the 5 g analysis.

15.2 High concentration method for soil samples with concentrations generally greater than 1 mg/kg.

15.2.1 The high concentration method for soil is based on a solvent extraction. A solid sample is either extracted or diluted, depending on sample solubility in a water-miscible solvent. An aliquot of the extract is added to organic-free reagent water containing surrogates and, if applicable, internal and matrix spiking standards. The sample is then purged according to Method 5030B, and analyzed by an appropriate determinative method. Wastes that are insoluble in methanol (i.e., petroleum and coke wastes) are diluted with hexadecane (see Sec. 15.2.10).

15.2.2 The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were <u>not</u> preserved in the field are prepared using the steps below,

beginning at Sec. 15.2.3. If solvent preservation was employed in the field, then the preparation begins with Sec. 15.2.6.

15.2.3 When the high concentration sample is not preserved in the field, the sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Whenever practical, mix the contents of the sample container by shaking or other mechanical means without opening the vial. When shaking is not practical, quickly mix the contents of the vial with a narrow metal spatula and immediately reseal the vial.

15.2.3.1 Samples received as specified in 15.2.3 will be preserved within 24 hours of receipt by the laboratory, provided the sample was not received late on Friday. When samples are received late on Friday, the samples will be preserved Monday morning. Sample preservation will be done following the procedure beginning in section 15.2.4.

15.2.4 If the sample is from an unknown source, perform a solubility test before proceeding. Remove several grams of material from the sample container. Quickly reseal the container to minimize the loss of volatiles. Weigh 1 g aliquots of the sample into several test tubes or other suitable containers. Add 10 ml of methanol to the first tube, 10 ml of PEG to the second, and 10 ml of hexadecane to the third. Swirl the sample and determine if it is soluble in the solvent. Once the solubility has been evaluated, discard these test solutions. If the sample is soluble in either methanol or PEG, proceed with Sec. 15.2.5. If the sample is only soluble in hexadecane, proceed with Sec. 15.2.10

15.2.5 For soil and solid waste samples that are soluble in methanol, add 9.0 ml of methanol and 1.0 ml of the surrogate spiking solution to a tared 20-ml vial. Using a top-loading balance, weigh 10 g (wet weight) of sample into the vial. Quickly cap the vial and re-weigh the vial. Record the weight to 0.1 g. Shake the vial for 2 min. If the sample was not soluble in methanol, but was soluble in PEG, employ the same procedure described above, but use 9.0 ml of PEG in place of the methanol. Proceed with Sec. 15.2.7.

NOTE: The steps in Secs. 15.2.3, 15.2.4, and 15.2.5 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

15.2.6 For soil and solid waste samples that were collected in methanol or PEG (see Sec. 8.2.3), add the surrogate spiking solution to the vial by injecting it through the septum, shake for 2 min., as described above, and proceed with Sec. 15.2.7.

15.2.7 After the soil has settled, pipet approximately 1 ml of the extract from either Sec. 15.2.5 or 15.2.6 into a GC vial for storage, using a disposable pipet, and seal the vial. The remainder of the extract may be discarded. Add approximately 1 ml of methanol or PEG to a separate GC vial for use as the method blank for each set of samples extracted with the same solvent.

15.2.8 The extracts must be stored at 4°C in the dark, prior to analysis. Add an appropriate aliquot of the extract (see Table 1) to 5.0 ml of organic-free reagent water and analyze by Method 5030B in conjunction with the appropriate determinative method.

15.2.9 If results are to be reported on a dry weight basis, the dry weight may be determined from a separate aliquot of the sample after the sample extract has been transferred to a GC vial and the vial sealed.

15.2.10 For solids that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste), dilute or extract the sample with hexadecane using the procedures in Method 3585.

15.3 High concentration method for oily waste samples

15.3.1 This procedure for the analysis of oily waste samples involves the dilution of the sample in methanol or PEG. However, care must be taken to avoid introducing any of the floating oil layer into the instrument. A portion of the diluted sample is then added to 5.0 ml of organic-free reagent water, purged according to Method 5030B, and analyzed using an appropriate determinative method.

15.3.2 For oily samples that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste), dilute or extract with hexadecane using the procedures in Method 3585.

15.3.3 The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were <u>not</u> preserved in the field are prepared using the steps below, beginning at Sec. 15.3.4. If methanol preservation was employed in the field, then the preparation begins with Sec. 15.3.6.

15.3.4 If the waste was <u>not</u> preserved in the field and it is soluble in methanol or PEG, weigh 1 g (wet weight) of the sample into a tared 10-ml volumetric flask, a tared scintillation vial, or a tared culture tube. If a vial or tube is used instead of a volumetric flask, it must be calibrated prior to use. This operation <u>must</u> be performed prior to opening the sample vial and weighing out the aliquot for analysis.

15.3.4.1 To calibrate the vessel, pipet 10.0 ml of methanol or PEG into the vial or tube and mark the bottom of the meniscus.

15.3.4.2 Discard this solvent, and proceed with weighing out the 1 g sample aliquot.

15.3.5 Quickly add 1.0 ml of surrogate spiking solution to the flask, vial, or tube, and dilute to 10.0 ml with the appropriate solvent (methanol or PEG). Swirl the vial to mix the contents and then shake vigorously for 2 minutes.

15.3.6 If the sample was collected in the field in a vial containing methanol or PEG, add the surrogate spiking solution to the vial by injecting it through the septum. Swirl the vial to mix the contents, shake vigorously for 2 minutes, and proceed with Sec. 15.3.7.

15.3.7 Regardless of how the sample was collected, the target analytes are extracted into the solvent along with the majority of the oily waste (i.e., some of the oil may still be floating on the surface). If oil is floating on the surface, transfer 1 to 2 ml of the extract to a clean GC autosampler vial using a Pasteur pipet. Ensure that no oil is transferred to the vial.

15.3.8 Add 10 - 50 ul of the methanol extract to 5 ml of organic-free reagent water for purge-and-trap analysis, using Method 5030B.

15.3.9 Prepare a matrix spike sample by adding 10 - 50 ul of the matrix spike standard dissolved in methanol to a 1 g aliquot of the oily waste. Shake the vial to disperse the matrix spike solution throughout the oil. Then add 10 ml of extraction solvent and proceed with the extraction and analysis, as described in Secs. 15.3.5 - 15.3.8. Calculate the recovery of the spiked analytes as described in appropriate method. If the recovery is not within the acceptance limits for the application, use the hexadecane dilution technique in Method 3585.

16.0 CALCULATIONS AND DATA HANDLING

16.1 See the determinative method for calculations and data handling.

17.0 DATA REPORTING AND DELIVERABLES

17.1 See the determinative method for data reporting and deliverable requirements.

18.0 QUALITY ASSURANCE

18.1 See the determinative method for quality assurance procedures.

19.0 ANALYST CERTIFICATION/METHOD VALIDATION

19.1 See the determinative method for analyst certification and method validation requirements.

20.0 REFERENCES

20.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Update III, Revision 3, December 1996, Methods 5035, 5030B, 8000B, 8021B, and 8260B.

Approved By: [signature] 4/30/98 — QA Manager

Approved By: [signature] 4/30/98 — Area Manager

21.0 ATTACHMENTS/APPENDICES

21.1 Table 1: Quantity of Methanol Extract Required for Analysis of High Concentration Soils/Sediments

Approved By: [signature] 4/30/98
QA Manager

Approved By: [signature] 4/30/98
Area Manager

TABLE 1
QUANTITY OF METHANOL EXTRACT REQUIRED FOR ANALYSIS OF HIGH CONCENTRATION SOILS/SEDIMENTS

Approximate Concentration Range	Volume of Methanol Extract[a]
0.50 - 10 mg/kg	100 ul
1 - 20 mg/kg	50 ul
5 - 100 mg/kg	10 ul
25 - 500 mg/kg	100 ul of 1:50 dilution[b]

Calculate appropriate dilution factor for concentrations exceeding those in this table.

a The volume of methanol added to 5 ml of water being purged should be kept constant. Therefore, add to the 5-ml syringe whatever volume of methanol is necessary to maintain a total volume of 100 ul of methanol.

b Dilute an aliquot of the methanol extract and then take 100 ul for analysis.

Approved By: 4/30/98 QA Manager

Approved By: 4/30/98 Area Manager

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

VOLATILE ORGANIC COMPOUNDS BY PURGE AND TRAP CAPILLARY COLUMN GAS CHROMATOGRAPHY/ MASS SPECTROMETRY

USEPA METHOD 8260B

USEPA METHOD 624

APPROVALS:

Organics Manager: _____ Date: 5/28/98
Jeff P. Glaser

QA Manager: _____ Date: 5/28/98
Rick D. Wilburn

Laboratory Manager: _____ Date: 5/28/98
Douglas E. Kriscunas

Procedure Number: GR-04-104
Revision Number: 3.0

Date Initiated: 9/1/95
Effective Date: 7/1/97

Date Revised: 5/28/98
Pages Revised: All

By: Jeff Glaser

Total Number of Pages: 56

1.0 SCOPE AND APPLICATION

1.1 Method 8260B/624 is a gas chromatograph/mass spectrometry (GC/MS) procedure for determining volatile organic compounds in a variety of matrices. This method can be used to quantitate most volatile organic compounds that have boiling points below 200°C. Volatile water-soluble compounds generally have higher quantitation limits due to poor purging efficiencies. Such compounds include low molecuLar weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides. It is applicable to nearly all type of samples, regardless of water content, including ground water, wastewater, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The sample is introduced into the system using a purge and trap concentrator or by direct injection.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Update III, Revision 2, December 1996, Method 8260B.

2.2 *Code of Federal Regulations, Title 40*, Part 136, Section 1, Appendix A, July 1, 1997, Methods 601 and 624.

3.0 SUMMARY OF PROCEDURE

3.1 When using the purge and trap technique, an inert gas is bubbled through the liquid sample at an ambient or slightly elevated temperature, and the volatile components are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column is heated and back flushed with inert gas to desorb the components onto a capillary column. The capillary column is temperature programmed to separate and elute the components, which are then detected with a mass spectrometer which provides qualitative or quantitative results.

3.2 Analytes are introduced into the mass spectrometer via a jet separator or a direct connection using an open-split interface. Identification of analytes is done by comparing their mass spectra with spectra of calibration standards. Quantitation is performed by comparing the response of the analytes quantitation ion (relative to its corresponding internal standard) with a five point calibration curve.

4.0 PARAMETER OR COMPOUND LIST

4.1 See Tables 1A and 1B.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP number GR-04-105, *Closed System Purge and Trap Extraction for Volatile Organics in Soil and Waste Samples*, most current version.

5.2 TriMatrix SOP number GR-3-124, *Volatile Laboratory Corrective Actions*, most current version.

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Whether purged or injected directly on column, interferences naturally present in the samples can cause elevated detection limits and interfere with the analysis. Naturally occurring interferences can vary considerably from site to site.

6.2 Sample contamination can also cause raised detection limits or false positives, and can come from a variety of sources. Improper sampling techniques can contaminate the sample at the job site. During shipment and storage, volatile organics (particularly methylene chloride and fluorocarbons) can diffuse through the septum and into the sample. A trip blank prepared from reagent water and carried through the sampling and handling protocol will serve as a check on such contamination.

6.3 During analysis, contamination can come from impurities in the purge gas, or from organic compounds outgassing from the plumbing ahead of the trap where they were deposited by a previously analyzed high level sample. To reduce this, the use of non-Teflon plastic tubing, non-Teflon thread sealants, and flow controllers with rubber components in the purging device have been eliminated where possible.

6.4 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe is rinsed with reagent water between samples. Where practical, samples with unusually high concentrations should be followed by an analysis of reagent water to check for cross contamination. If the compounds present in high concentrations are also present is subsequent samples, the analyst must demonstrate that the positive results are not due to carryover by reanalysis of the samples. However, if the compounds are not present in the subsequent sample, then reanalysis is not necessary.

6.5 The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required. If the contamination is persistent, the complete purge and trap system should be purged first with 100°C reagent water, then, if necessary, with methanol. If methanol is used, either disconnect the trap or install a blank trap, as the methanol will adversely affect the trap's performance.

6.6 For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high organohalide levels, it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105°C oven between analyses.

6.7 When using an ion trap, methanol content in blanks, standards, and samples should be kept as constant as possible because varying amounts of methanol can cause different spectra for certain compounds. (Chloroethane spectra does not look like reference spectra due to this phenomenon). **Maximum amount of methanol to be purged is 100 uL.**

7.0 SAFETY PRECAUTIONS

7.1 The analyst must comply with all standard operating procedures for health and safety as outlined in the "TRIMATRIX Safety Manual".

7.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material handling data sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified for the information of the analyst.

7.3 The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichloroethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromomethane, tetrachloroethene, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Sample Collection

8.1.1 Collect all aqueous and liquid waste samples in duplicate 40 ml borosilicate glass screw-cap VOA vials with teflon lined silicone septa. Gently fill sample bottles to overflowing to minimize loss of volatiles. No air bubbles should pass through the sample as the bottle is filled, or be trapped in the sample when the bottle is sealed. Invert the vial the confirm that

there is no headspace present. If there is headspace or bubbles larger than 5-6 mm in diameter, refill a new preserved vial.

8.1.2 Collect soil/sludge/solid waste samples in accordance with USEPA Method 5035, as specified in TriMatrix SOP number GR-04-105 *Closed System Purge and Trap Extraction for Volatile Organics in Soil and Waste Samples*, most current version.

8.2 Sample Preservation

8.2.1 Aqueous Samples

8.2.1.1 For aqueous samples, if the sample contains residual chlorine, collect the sample in a 125-ml container which has been pre-preserved with 4 drops of 10% sodium thiosulfate solution. Gently swirl to mix sample and transfer to a 40 ml VOA vial. Cool to 4°C and adjust pH to <2 with HCl. EPA Methods 330.4 and 330.5 may be used for measurements of residual chlorine. Field test kits are also available for this purpose.

8.2.1.2 TriMatrix Laboratories supplies all clientele with 40 ml VOA vials which are pre-preserved with HCl. If the vials were not supplied by the laboratory, and are not pre-preserved, fill vials and adjust the pH to <2 by carefully adding two drops of 1:1 HCl to each sample. Seal the sample bottle, PFTE-face down, and cool to 4 °C.

8.2.1.3 Samples collected for Acrolein and/or Acrylonitrile are to be preserved at a pH of 4-5 with 1:1 HCl or analyzed within 3 days of collection.

8.2.2 Soil/Sludge/Waste Samples

8.2.2.1 See section 8.1.2.

8.3 Trip Blanks - Aqueous Samples Only

8.3.1 A trip blank should accompany each sample set into the field and back. A sample set is composed of the samples collected from the same general sample site at approximately the same time. At the laboratory, fill a pre-preserved sample bottle with reagent water, seal, and ship to the sampling site along with empty sample bottles. Wherever a set of samples is shipped and stored, it is accompanied by the appropriate trip blank.

8.4 Sample Storage

8.4.1 The samples must be chilled to 4°C on the day of collection and maintained at that temperature until analysis. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure that they will be at 4°C on arrival at the laboratory.

8.4.2 Store samples at 4°C until analysis. The laboratory sample storage area must be free of organic solvent vapors. Soils/sludges/and wastes are stored separately from aqueous samples.

8.4.3 Analyze all samples within 14 days of collection. Samples not analyzed within this period must be qualified as estimated.

8.4.4 If aqueous samples are unpreserved, hold time for aromatics is 7 days. Hold time for acrolein is 3 days.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Glassware and Hardware

9.1.1 Various size class A volumetric flasks. (10 mL, 50 mL, 100 mL, 250 mL, 1000 mL)

9.1.2 Various size microsyringes. (1 uL, 5 uL, 10 uL, 25 uL, 50 uL, 100 uL, 1000 uL)

9.1.3 5 mL gas tight luerlock syringes

9.1.4 60 or 125 mL wide mouth with Teflon-lined cap.

9.1.5 Borosilicate glass vials with teflon lined septum caps, 20 ml and 40 mL.

9.1.6 Various size teflon lined screw cap vials

9.1.7 refrigerator

9.1.8 pH test strips

9.1.9 metal spatulas

9.1.10 analytical balance 0.0001 g & top-loading balance - 0.01 g.

9.1.11 pasteur pipets (disposable) with rubber pipet bulb

9.2 Instrumentation

9.2.1 Concentrators (Other concentrators may be substituted if equivalent data can be produced.)

9.2.1.1 Dynatech Precision Sampling Dynasoil concentrator recommended conditions:

Trap: Supelco Vocarb 4000
preheat: 1 min. at 40°C
purge: 11 min. at 35-40 mL/min.
dry purge: 3 min.
desorb preheat: 225°C
desorb: 230°C for 2.0 min.
bake: 8 min. at 250°C
water trap: low = 38°C, high = 150°C
valve oven: 125°C
heated transfer line: 125°C

9.2.1.2 Tekmar LSC-2000 conentrator recommended conditions:

Trap: Supelco Vocarb 4000 (Carbopack/Cabosieve may also be used)
preheat: 1.0 min at 40°C
purge: 11 min. at 35-40 mL/min.
dry purge: 3 min.
desorb preheat: 245°C
desorb: 250° C for 2.0 min.
bake: 8 min. at 260°C
valve oven: 125°C
heated transfer line: 125°C

9.2.2 Autosamplers

- O. I. Analytical 4552 (Archon)
- Tekmar ALS 2016

- Dynatech PTA 30 W/S

9.2.3 Gas Chromatograph

9.2.3.1 Varian model 3400 temperature programmable gas chromatograph or equivalent equipped with a SPI injector and an Open Split Interface (OSI). Recommended conditions:

Injector Temperature: 150°C
Transfer Line Temperature: 250°C
OSI Temperature: 250°C
OSI Split Ratio: 20:1

Temperature Program: 10°C for 1 minute, then to 150°C at 6°C/min, then to 200°C at 15°C/min, hold at 200°C for 4-5 minutes.

9.2.3.2 Hewlett Packard Model 5890 Series II with a low dead volume injection port and glass jet separator. Recommended conditions:

Injector Temperature: 200°C
Transfer Line Temperature 280°C
Jet Separator Temperature: 250°C

Temperature Program: 35°C for 10 minutes, then 5°/min to 110°C, hold 1 minute, then 20°/min to 220°C, hold 2 minutes.

9.2.3.3 Hewlett Packard Model 5890 Series II equipped with electronic pressure control (EPC), a split/splitless injection port and a capillary direct interface.

Injector Temperature: 180°C
Transfer Line Temperature 280°C
EPC setting: Constant flow mode at 1.0 ml/min
Split Ratio: 40:1

Temperature Program: 35°C for 10 minutes, then 7°/min to 73°C, hold 1 minute, then 10°/min to 110°C, hold 1 minute, then 20°/min to 220°C, hold 2 minutes.

9.2.4 Columns:

9.2.4.1 75m x 0.53 mm ID, 3.0 um film thickness wide bore capillary column (J&W Scientific DB-624 or equivalent). Used in Varian 3400 (9.2.3.1).

9.2.4.2 75 m x 0.45 mm ID, 2.5 um film thickness, wide bore capillary column DB-VRX (J&W Scientific). Used in HP 5890 II with jet separator (9.2.3.2).

9.2.4.3 30 m x 0.25 mm ID, 1.4 um flim thickness, narrow bore capillary column DB-VRX (J&W Scientific). Used in HP 5890 II with capillary direct interface (9.2.3.3).

9.2.5 Mass Spectrometer (Saturn Ion Trap or Hewlett Packard 5971 MSD) capable of scanning from 35-650 amu every 3 seconds or less, using 70 volts (nominal) electron energy in the electron impact mode and producing a mass spectrum that meets all the criteria in Table 3 when 50 ng of 4-bromofluorobenzene (BFB) are purged onto the analytical column.

GC/MS recommended operating conditions:
Electron energy: 70 volts (nominal).
Mass range: 38-400 amu.
Scan time: 359 amu/second = 1 scan/sec.
Source temperature: 160 - 220°C

9.2.6 Data Acquisition

9.2.6.1 The Saturn data system is a DOS-based system that allows continuous acquisition and storage of all results obtained throughout the duration of each acquisition on hard disk. The software is able to process the data file and plot the ion abundance's of a specified mass versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). The data files are then converted from Saturn files to HP files and processed using HP Enviroquant, an emvironmental data analysis software. The software also has a 75,000 compound NBS spectral library used in identifying unknown compounds.

9.2.6.2 The HP MSD data acquisition system is DOS-based HP Chemstation equipped with Enviroquant, an environmental data analysis software. It is also capable of plotting EICP's and has a 75,000 compound NBS spectral library.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 GC Maintenance

10.1.1 Change septa weekly or as needed.

10.1.2 Change gas scrubber traps yearly or as needed.

10.1.3 Clip or replace GC column as needed.

10.1.4 Check column head pressure daily.

10.1.5 Check gas cylinder pressure daily and change if needed.

10.2 Mass Spec Maintenance

10.2.1 Ion Trap Maintenance Change

10.2.1.1 Mechanical pump oil every 6 months.

10.2.1.2 Clean Ion Trap Analyzer at least annually.

10.2.1.3 Replace prefilled oil reservoir every 6 months.

10.2.1.4 Examine o-ring gasket seals and replace if necessary after analyzer maintenance.

10.2.1.5 Replace the electron multiplier as necessary

10.2.2 MSD Maintenance

10.2.2.1 Change rough pump oil every six months.

10.2.2.2 Check diffusion pump oil annually and change if discolored or low.

10.2.2.3 Clean ion source at least annually (maybe needed more frequently as shown by detector performance).

10.2.2.4 Examine & replace large o-ring seal if worn.

10.2.2.5 Replace electron multiplier as needed. (Maximum voltage 3000).

10.3 Purge and Trap

10.3.1 Bake out trap daily before use

10.3.2 Check purge flow weekly

10.3.3 Empty waste bottles and fill rinse bottles daily

10.3.4 Check concentrator pressures

10.3.5 Check and fill internal/surrogate standard syringe daily

11.0 CHEMICALS AND REAGENTS

11.1 Chemicals

11.1.1 Reagent water (organic free) ASTM Type II

11.1.2 Methanol (purge and trap grade)

11.1.3 Pure stock standard materials or certified stock standards (96% pure or greater)

11.1.4 1:1 Hydrochloric Acid

11.1.5 Polyethylene glycol - Free of interferences at the detection limit of the target analytes.

11.1.6 Sodium bisulfate, Na_2HSO_4, ACS reagent grade or equivalent.

12.0 STANDARDS PREPARATION

12.1 All standards used in the laboratory must be recorded in the standards logbook. Vendors, lot #'s, concentrations, purity's, etc. are recorded. Each standard is given a unique ID #.

12.2 Stock & Intermediate Standard Storage Requirements

12.2.1 Gas standards should be monitored closely for degradation. Stock standards for gases usually will need to be replaced after month. Intermediate gas standards usually will need to be replaced after one week. If the continuing calibration standard varies by more than 25% from the initial calibration curve, then a new gas standard needs to be prepared. The first compound to fail will generally be dichlorofluoromethane, so it's response should be monitored closely.

12.2.2 All other stock standards may be kept up to 12 months. Intermediate standards may be kept for up to 6 months; however, they should be continuously monitored for degradation by comparison of the response of the compounds in daily continuing calibration standards to that of the initial calibration curve.

12.2.3 2-Chloroethyl vinyl ether should be prepared separately from the other compounds due to its reactivity. Intermediate standards should be prepared fresh monthly.

12.2.4 All stock and intermediate standards are to be stored with minimal headspace at -10°C to -20°C.

12.3 Stock Standards

12.3.1 Stock Solutions may be prepared from pure standard materials or purchased as certified solutions. Commercially prepared stock standards can be used a[illegible]ny concentration if they are certified by the manufacturer or by an independent source.

12.3.2 If preparing stock standards from neat compounds no adjustment factor is needed when the chemical has greater than or equal to 96% purity. If the purity is less than 96% then an adjustment factor must be used. Prepare stock standard solution in methanol using assayed liquids. Transfer the stock standard solution into teflon sealed screw-cap bottles. Store in a freezer separate from samples and protect from light.

12.3.2.1 Gravimetric Method
10,000 mg/L stock standard: 0.5g of each analyte (neat) is injected into a 50 mL volumetric flask partially filled with methanol which has been tared on an analytical balance. The weight is recorded to the nearest 0.0001g the volumetric filled to the mark with methanol, capped and inverted three times to ensure proper mixing.

12.3.3 Commercial Stock Standards are purchased as custom mixtures from Accustandard Inc.

12.3.3.1 Standard IA: Volatile Organic Compounds - Liquids @ 2.0mg/mL in MeOH (Accustandard M502A-R-10X). See Table 2A.

12.3.3.2 Standard IB: Volatile Organic Compounds - Gases @ 2.0mg/mL in MeOH (Accustandard M502B-10X). See Table 2A.

12.3.3.3 Standard IC: Acrolein & Acrylonitrile @ 1.0 mg/mL in water (Accustandard M-603/M-001M).

12.3.3.4 Standard ID: 2-Chloroethyl Vinyl Ether @ 2.0 mg/mL in MeOH (Accustandard M-601C-10X).

12.3.3.5 Standard IE: Custom Additions to Method 8260 @ 2.0 mg/ml in MeOH (Accustandard S-3439-R1). See Table 2A.

12.3.3.6 Standard II: Appendix IX Standard @ 2.5 mg/mL in MeOH (Accustandard S-3651 Volatile Custom Solution). See Table 2B.

12.3.3.7 Standard III: Internal Standard/Surrogate Standard Mixture: These standards are purchased commercially at 2.0 mg/ml for the Ion Trap and at 5.0 mg/ml for the MSD's. The compounds are also different between the two instruments due to spectral interferences on the Ion Trap for the recommended 8260B internal and surrogate standards.

12.3.3.7.1 Internal Standards for Ion Trap (Ultra Scientific

12.3.3.7.1.1 Pentafluorobenzene

12.3.3.7.1.2 1,4-Difluorobenzene

12.3.3.7.1.3 Chlorobenzene-d5

12.3.3.7.1.4 1,4-Dichlorobenzene-d4

12.3.3.7.2 Surrogate Standards for Ion Trap (Ultra Scientific

12.3.3.7.2.1 Dibromofluoromethane

12.3.3.7.2.2 Toluene-d8

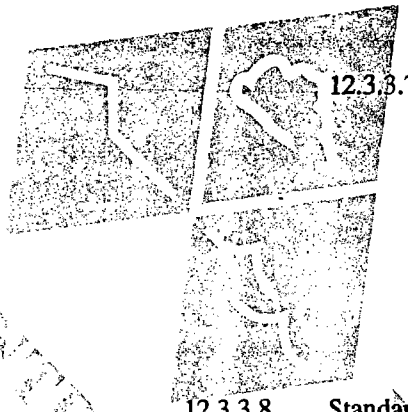
12.3.3.7.2.3 4-Bromofluorobenzene

12.3.3.7.3 Internal Standards for HP 5971 MSD (Ultra Scientific

12.3.3.7.3.1 Fluorobenzene

12.3.3.7.3.2 Chlorobenzene-d5

12.3.3.7.3.3 1,4-Dichlorobenzene-d4



12.3.3.7.4 Surrogate Standards for HP 5971 MSD (Ultra Scientific

12.3.3.7.4.1 Dibromofluoromethane

12.3.3.7.4.2 1,2-Dichloroethane-d4

12.3.3.7.4.3 Toluene-d8

12.3.3.7.4.4 4-Bromofluorobenzene

12.3.3.8 Standard IV: Standard Surrogate @ 2.0 mg/mL in MeOH (Ultra Scientific STM-330N).

12.4 Intermediate Standards - concentrations of working, surrogate, and internal standards are recommended and subject to change.

12.4.1 8260 Intermediate Standard: This standard will be at a concentration of 100 ug/mL. To make the 100 ug/mL standard add 50 uL of IA (12.3.3.1), IB (12.3.3.2), ID (12.3.3.4), and IE (12.3.3.5) to 800 uL of methanol.

12.4.2 Acrolein and Acrylonitrile Intermediate Standard: This standard will be at a concentration of 100 ug/mL. To make the 100 ug/mL standard, add 100 uL of IC (12.3.3.3) to 900 uL of methanol.

12.4.3 Appendix IX Intermediate Standard: This standard will be at a concentration of 250 ug/mL. To make the 250 ug/mL standard, add 100 uL of II (12.3.3.6) to 900 uL of methanol.

12.4.4 Surrogate Intermediate Standard A: This standard will be at a concentration of 50 ug/mL. To make the 50 ug/mL standard, add 25 uL of IV (12.3.3.8) to 975 uL of methanol. This standard will be used for the initial calibration curves.

12.4.5 Surrogate Intermediate Standards B: This standard will be at a concentration of 20 ug/ml. To make the 20 ug/ml standard, add 10 uL of IV (12.3.3.8) to 990 uL of methanol. The surrogate standard will be used for spiking methanol extracted samples.

12.4.6 Combined Intermediate Internal Standard/Surrogate Standard Mixture

12.4.6.1 If adding IS/SS standard manually, make a working standard by adding 1 mL of a 2.0 mg/mL commercially prepared standard III into a 3/4 full 100 mL volumetric flask. Add the standard directly to the methanol to prevent any loss of the volatile

compounds. Follow the same procedure for the commercially prepared surrogate standard solution. Dilute to volume with methanol, stopper, and invert 3 times. This will produce a working standard at 20 ug/mL. Transfer the solution to as many 1.5 mL auto sampler vials as possible and store in the freezer.

12.4.6.2 If the purge and trap auto sampler will add the IS/SS Standard automatically, prepare a working standard solution at 200 ug/mL by diluting 1 mL of the 5.0 mg/ml commercially prepared standard III into a 3/4 full 25 mL volumetric flask and dilute to volume. Store in the freezer.

12.5 Calibration and Calibration Check Standards are prepared in water in either a 50 mL volumetric flask or a 5 mL gas-tight syringe using the formula below. CCV standards are at 40 ug/L for all compounds excpet the Appendix IX compounds which are at 100 ug/L.:

12.5.1 Working standards are prepared in water in either a 50 mL volumetric or a 5 mL syringe using the formula below:

$$Vs = \frac{Cf\ Vf}{Cs}$$

where:

Vs = volume of stock to inject (uL)
Cf = final concentration of working std (ug/L)
Vf = final volume of working stds (uL)
Cs = concentration of stock (ug/L)

These Standards are prepared as needed and are not stored. See section 14.2.2

12.6 Matrix Spike Standards

12.6.1 Intermediate stock standards as prepared in section 12.4.1 are used for matrix spiking purposes. Matrix spikes are prepared using the formula given in section 12.5. The final concentration will be 40 ug/L.

12.6.2 For a quality control purposes, the following compounds are routinely reported: 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene.

12.6.3 **For the State of Wisconsin samples, all analytes from the matrix spike standard are to be reported and compared to statistical control limits.**

12.7 Laboratory Fortified Blank (LFB) Standards

12.7.1 Intermediate stock standards as prepared in section 12.4.1 are generally used for laboratory fortified blanks. LFBs are prepared using the formula given in section 12.5. The final concentration will be 40 ug/L.

12.7.2 For routine quality control purposes, the following compounds are routinely reported: 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene.

12.7.3 **For the State of Wisconsin samples, all analytes from the LFB standard are to be reported and compared to statistical control limits.**

12.8 Laboratory Control Sample (LCS) Standards

12.8.1 Intermediate stock standards as prepared in section 12.4 are used for Laboratory Control Samples. The analyst must use a different lot # standard than the one used for the calibration standards. The LCS standards are prepared as in 12.5 and are made using standards IA, IB, IC, ID and IE for a regular 8260B curve or using standard II for an appendix IX 8260B curve.

12.8.2 All initial calibration curve analytes are spiked into the LCS standard. For a complete list see Tables 1A and 1B. The concentrations are 40 ug/L for regular 8260 and 100 ug/L for Appendix IX.

13.0 SAMPLE PREPARATION

This section includes procedures beginning with removing sample from refrigeration until sample has been concentrated onto the sorbent trap.

13.1 Allow the sample to come to ambient temperature prior to introducing it to the syringe. If either the Dynatech PTA 30 or O.I. 4552 autosampler will be used for sample analysis, the sample will need no preparation - the 40 ml vial will be loaded directly into the autosampler. I. the ALS 2016 or Dynasoils autosamplers will be used, remove the plunger from a 5 ml syringe. Open the sample bottle and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger. Vent any residual air while adjusting the sample volume to 5.0 ml. Since this process of taking an aliquot destroys the validity of the sample for future analysis, if only one sample vial has been provided, the analyst should immediately place the remaining sample in a 20 ml vial with no headspace, to be used if subsequent analysis is necessary. Pull back slightly on the plunger, spike 10 ul of the internal/surrogate standard solution (12.4.6.1) directly into the 5 ml syringe, and inject the sample into purging chamber.

13.1.1 For matrix spikes, if using Tekmar instruments, add 2 ul of the 100 ug/ml intermediate stock standard (12.4.1) in addition to the IS/SS standard If using a Dynatech or O.I. instrument, add 26 ul of the 100 ug/ml intermediate stock standard directly to a 40 ml vial. Refer to section 9.2.2 for recommended purge and trap conditions. Spiking amounts are subject to change.

13.2 Soil/Sludge and Waste Samples:

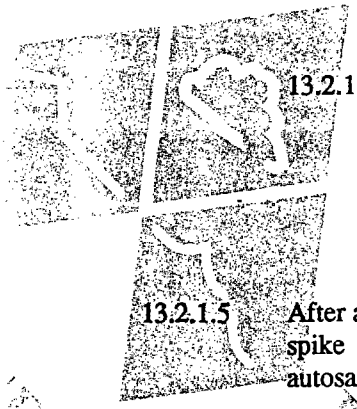
13.2.1 Low Concentration Method: This is designed for samples containing individual purgeable compounds of <1 mg/kg. TriMatrix employs two methods for the low-level soils. The closed system purge and trap by EPA method 5035 is based on purging a heated 5 gram sample collected in a vial pre-preserved with a sodium bisulfate solution. This procedure is described in detail in TriMatrix SOP GR-04-105. The other method, based on the older 5030 method preparation is detailed below. It is based on purging a heated sample mixed with organic-free reagent water containing the internal/surrogate standard and, if applicable, matrix spiking standards. Analyze all blanks, spikes, standards, and samples under the same conditions as the samples.

13.2.1.1 Use a 5g sample if the expected concentration is <0.1 mg/kg or a 1g sample for expected concentrations between 0.1 and 1 mg/kg.

13.2.1.2 A heated purge calibration curve must be prepared and used for the quantitation of all samples analyzed with the low concentration method. Follow the initial and daily calibration instructions, except for the addition of a 40° purge temperature.

13.2.1.3 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh the amount determined in Section 13.2.1.1 into a tared purge device or autosampler vial. Note and record the actual weight to the nearest 0.1 g.

13.2.1.4 If a Dynatech PTA 30 or O.I. 4552 autosampler is to be used, then weigh out the soil directly into an autosampler vial. The autosampler will automatically add 5 ml of water, along with 1 ul of the IS/SS standard (12.4.6.2) to the sample container before purging. If a Tekmar ALS 2016 is to be used, remove the plunger from a 5 ml Luerlock type syringe and fill until overflowing with organic-free reagent water. Replace the plunger and compress the reagent water to vent trapped air. Adjust the volume to 5.0 ml. Pull back slightly on the plunger, and spike 10 ul of the IS/SS standard solution (12.4.6.1) directly into the 5 ml syringe.



13.2.1.4.1 For matrix spikes, if using Tekmar instruments, add 2 ul of the 100 ug/ml intermediate stock standard (12.4.1) in addition to the IS/SS standard If using a Dynatech or O.I. instrument, add 26 ul of the 100 ug/ml intermediate stock standard directly to a 40 ml vial. Refer to section 9.2.2 for recommended purge and trap procedures. Spiking amounts are subject to change.

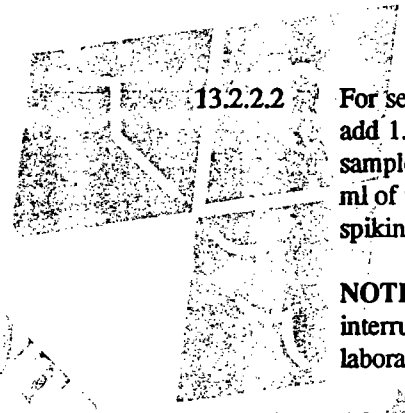
13.2.1.5 After addition of the reagent water containing the IS/SS standard (and any matrix spike standards, if necessary), load the sample onto the purge and trap autosampler system.

NOTE: Prior to the attachment of the purge device, Sections 13.2.1.4 and 13.2.1.5 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

13.2.1.6 Fit the heating jacket over the purging device if using a Tekmar heated purge and trap unit. (The Dynatech autosampler uses a heated purging chamber.) Heat the sample to 40°C while purging. Refer to section 9.2.2 for recommended purge and trap procedures.

13.2.2 High Concentration Method: The method is based on extracting the soil/sludge with methanol. A waste sample is either extracted or diluted, depending on its solubility in methanol. Wastes (i.e., petroleum and coke wastes) that are insoluble in methanol are diluted with polyethylene glycol (PEG). An aliquot of the extract is added to organic-free reagent water. This is purged at 40°C. All samples with an expected concentration of >1.0 mg/kg should be analyzed by this method.

13.2.2.1 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquius. Mix the contents of the sample container with a narrow metal spatula. For sediment/soil and waste that are insoluble in methanol, weigh 4g (wet weight) of sample into a tared 20 ml vial. Use a top loading balance. Note and record the actual weight to 0.1 gram. For waste that is soluble in methanol or PEG, weigh 1g (wet weight) into a tared scintillation vial or a 10 ml volumetric flask. (If a vial or tube is used, it must be calibrated prior to use. Pipet 10.0 ml of the appropriate solvent into the vial and mark the bottom of the meniscus. Discard this solvent.) Other solvents and procedures are covered in TriMatrix SOP number GR-04-105, *Closed System Purge and Trap Extraction for Volatile Organics in Soil and Waste Samples*



13.2.2.2 For sediment/soil or solid waste, quickly add 9.0 ml of appropriate solvent; then add 1.0 ml of the surrogate standard (12.4.5) to the vial. For a solvent miscible sample, dilute the sample to 10 ml with the appropriate solvent after adding 1.0 ml of the surrogate standard (12.4.5). For matrix spikes, add 200 ul of the matrix spiking standard (12.4.1). Cap and shake for 2 min.

NOTE: Sections 13.2.2.1 and 13.2.2.2 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

13.2.2.3 After settling has occurred, transfer approximately 4 ml of the extract into a vial for storage, using a disposable pipet. The remainder may be discarded. These extracts should be stored at 4°C in the dark, prior to analysis.

13.2.2.4 For each batch of samples prepped the analyst must also prepare a Method Preparation Blank (MPB) and a Laboratory Fortifier Blank (LFB). For the MPB, add 1.0 ml of the surrogate standard (12.4.5) to 9.0 ml of the appropriate solvent. For the LFB, use 0.2 ml of the matrix spiking(12.4.1) in addition to the surrogate standard and 8.0 ml of solvent.

13.2.2.5 If a screening procedure was followed, use the estimated concentration to determine the appropriate volume of methanol extract to add to the 5 ml of organic-free reagent water for analysis. Otherwise, estimate the concentration range of the sample from the low concentration analysis to determine the appropriate volume. If the sample was submitted as a high concentration sample, start with 100 ul. All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

13.2.2.6 Remove the plunger from a 5.0 ml Luerlock type syringe and fill until overflowing with organic-free reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 ml. Pull the plunger back to 5.0 ml to allow volume for the addition of the sample extract. Add the volume of methanol extract determined in Section 13.2.2.5 and a volume of methanol solvent to total 100 ul (excluding methanol in standards). Inject 10 ul of internal standard (12.3.3.7.1). Inject sample into purging chamber. Refer to section 9.2.2 for recommended purge and trap procedures.

13.2.2.7 Analyze the MPB, LFB, and duplicate matrix spikes as in section 13.2.2.6.

14.0 CALIBRATION PROCEDURES

14.1 Tuning Criteria

14.1.1 No analysis of any kind may begin until 50 ng of 4-Bromofluorobenzene (BFB) injected onto the system produces a spectra that passes the tuning criteria given in Table 3.

14.1.2 The internal standard/surrogate mix as prepared in 12.4.6.1 contains 20 mg/L of 4-Bromofluorobenzene (BFB). Purging 2.5 uL of this standard in 5 mLs of reagent water puts 50 ng of BFB on column. BFB's must be the initial run every 12 hours for method 8260 and every 24 hours for method 624 before any samples, blanks or standards are run.

TABLE 3 - BFB KEY ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

14.2 Initial Calibration

14.2.1 In addition to passing the BFB tuning criteria, a 5 point calibration curve must initially be run before any samples can be analyzed. A heated curve is required if low level heated soils are to be analyzed. **TriMatrix analyzes all standards and samples using a heated purge.** All target compounds and surrogates must be included in the 5 point curve including internal standards and surrogates.

14.2.2 For Dynasoils and Tekmar-ALS 2016 Autosamples remove the plunger from a 5 mL luer lock syringe. Fill the syringe with reagent water. Replace the syringe plunger and compress the sample. Tilt the syringe so the tip is in the air and vent any residual air while adjusting the sample volume to 5.0 mL. Pull back slightly on the plunger to create a small head space. Add 10 uL of internal standard standard spiking solution (12.4.6.1) through the tip of the syringe. Next, using a syringe, withdraw the volumes of standards specified in Tables 4A or 4B and add the aliquot directly to the 5 mL syringe through the tip. When discharging the contents of the

micro syringe, be sure that the end of the syringe needle is well beneath the surface of the reagent water in the 5 mL syringe. Attach the syringe to the syringe valve on the purging device. Open the valve and inject the sample into the purging chamber. Close the valve. Start purging and data acquisition. Other concentration levels than those in Tables 4A and 4B may be analyzed. Note: a constant amount of internal standard is added to all standards, while surrogate concentration will vary. The second number in table 4A is the surrogate standard concentration in each standard.

14.2.3 For the Dynatech PTA 30 W/S Autosampler, prepare standards in 50 mL volumetric flasks using 10 times the amounts listed in Table 4 A&B. Transfer the sample to a 40 mL vial and load on the autosampler. The autosampler will take a 5 mL aliquot and add 1 uL of internal surrogate standard mixture before purging.

For soil samples and heated water samples, follow the same procedure only that a 5 mL aliquot of standard must be manually added to a 40 mL vial. Internal/surrogate standard will be automatically added before purging. All heated standards will be heated at 40°C.

14.2.4 Calculate Response Factors (RF's) for each compound using the quantitation ion, and the internal standard ion listed in Tables 5A or 5B. The RF is calculated as follows:

$$RF = \frac{A_x * C_{is}}{A_{is} * Cx}$$

where:
Ax = Area of the characteristic ion for the target compound.
Ais = Area of the characteristic ion for the specific internal standard.
Cx = Concentration of the compound being measured.
Cis = Concentration of the specific internal standard.

TABLE 4A
STANDARD 8260 VOLUMES REQUIRED FOR CURVE

Concentration of Working Std (ug/L 8260/Surr)	Volume (uL) of Standard (12.4.1)	Volume (uL) of Standard (12.4.2)	Volume (uL) of Standard III (12.4.6.1)	Volume (uL) of Standard IV (12.4.4)
10/80	0.5	0.5	10.0	4.0
20/70	1.0	1.0	10.0	3.0
40/40	2.0	2.0	10.0	---
100/60	5.0	5.0	10.0	2.0
200/50	10.0	10.0	10.0	1.0

*Note: For standard made-up 50 mL volumetric flasks, add 10 times the above amounts.

TABLE 4B
APPENDIX IX VOLUMES REQUIRED FOR CURVE

Concentration of Working Std (ug/L)	Volume (uL) of Standard (12.4.3)	Volume (uL) of Standard III (12.4.6.1)
25	0.5	10.0
50	1.0	10.0
100	2.0	10.0
250	5.0	10.0
500	10.0	10.0

Approved By: R+ 5/28/98 QA Manager

Approved By: JA 5/28/98 Area Manager

14.2.5 For compounds not listed in Tables 5A or 5B, a major ion that is dissimilar to potential coeluting or interfering ions should be chosen as the quantitation ion. The internal standard chosen should be one that has a retention time closest to the compound being measured.

14.2.6 An average RF for all 5 points of the curve must be calculated for each compound. Five compounds are checked for a minimum average response factor. These are the System Performance Check Compounds (SPCC's). They are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. The minimum relative response factor for the SPCC's are as follows:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	>0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

This check is used to monitor compound instability and degradation caused by contaminated lines or active sites in the system. Examples of these occurrences are:

14.2.6.1 Chloromethane: This compound is the most likely compound to be lost if the purge flow is too fast.

14.2.6.2 Bromoform: This compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio may improve bromoform response.

14.2.6.3 Tetrachloroethane and 1,1-dichloroethane: These compounds are degraded by contaminated transfer lines in purge and trap systems and/or active sites in trapping materials.

14.2.7 The Percent Relative Standard Deviation (% RSD) also must be calculated for all compounds. Using the RF's from the initial calibration, calculate the % RSD using the following formula:

$$\% \text{ RSD} = (SD/x) * 100$$

where

RSD = relative standard deviation.
x = mean of the five initial response factors for a compound.

SD = standard deviation of average RF's for a compound.

All compounds should have a % RSD of ≤ 15%. Six compounds are used as Calibration Check Compounds (CCC's): 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethyl benzene, vinyl chloride. These compounds must have a % RSD of ≤30%. If the % RDS is greater than 30 percent for any CCC, corrective action must be initiated and the system must be recalibrated.

14.2.8 If the % RSD of an non-CC compound is 15% or less, the relative response factor is assumed constant over the calibration range, and the average relative response factor may be used for quantitation.

14.2.9 If the % RSD of any non-CCC compound is greater than 15%, than the analyst must construct calibration curves of area ratio (Area analyte/Area-IS) verses concentration using first or higher order regression fit of the five calibration standards. The analyst should select the option which provides the least calibration error.

14.3 Continuing Calibration

14.3.1 Every 12 hours a 40 ug/L continuing calibration standard containing each compound being quantitated is run after the BFB (see section 14.1). If a calibration curve has just been run then the 40 ug/L standard from the curve will be used as the 12 hour standard. This is done to verify instrument sensitivity, and to confirm the initial calibration curve is still valid. The continuing calibration standard is verified similarly to the initial calibration curve, by checking the SPCC's and the CCC's. The run must be heated if performing low level heated soil analyses.

14.3.1.1 System Performance Check Compounds (SPCC's): This is the same check that is applied during the initial calibration. The response factor for the SPCC compounds must be as listed in 14.2.6. (See section 14.2.4 for response factor calculation.) If these minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

14.3.1.2 Calibration Check Compounds (CCC's): After the SPCC's are met, the CCC's are used to check the validity of the initial calibration. Calculate the percent drift using the following formula:

$$\% \text{ Drift} = (C_1 - C_c)/C_1 \times 100$$

where:

C_1 = Calibration Check Compound Standard concentration.
C_c = Measure concentration using selected quantitation method.

If the % Drift for each CCC is less than 20%, the initial calibration is assumed to be valid. If the criterion is not met then corrective action must be taken. This criterion **MUST** be met before sample analysis can begin. If the CCC compounds are not analytes of interest, then all target analytes must adhere to the less than 20% criteria.

14.3.1.3 The internal standard responses and retention times of the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds as compared to the 40 ug/L standard, the system must be inspected for malfunctions and corrections must be made as needed. If the area for any of the internal standards changes by a factor of two (-50% to + 100%) as compared to the 40 ug/L standard, the system must be inspected for malfunctions and corrections must be made as needed. When corrections are made, reanalysis of the samples analyzed while the system was malfunctioning are necessary.

13.4 Blank

13.4.1 After the BFB and the curve or continuing calibration has been run, a reagent water blank is required before any samples are analyzed showing that the analytical system is free from interferences and contaminaiton. The acceptance criteria for the instrument blank is that all analytes of interest must be below their minimum reporting limits for all samples to be analyzed in that analtyical batch, except for common lab contaminants (methylene chloride or acetone), which may be 5X the reporting limit.. At a minimum a blank is run every 12 hour shift. A blank will be run more frequently if contamination is suspected from a high level sample or if laboratory contamination is in question. The blank samples should be carried through all stages of the sample preparation and measurement steps, and will be spiked with internal standards and surrogates.

15.0 ANALYTICAL PROCEDURE

15.1 Sample Analysis

Desorb the sample onto the column which should be temperature programmed to separate the analytes of interest. Refer to section 9.2.1, 9.2.2, and 9.2.3 for recommended desorb, bake and column temperature settings.

15.2 If the response for any compounds exceeds the working range of the calibration curve, prepare a dilution of the sample.

15.2.1 Water Samples: Prepare a dilution of the sample from the aliquot in the 20 mL vial (or a second vial) in either a 5 mL syringe or a volumetric flask. Total volume purged must be 5 mL. Organic free water must be used for the dilution. Add 10 uL of the internal standard/surrogate spike mix (12.4.6.1) to the syringe. Inject into purge device and start purging and data acquisition. Alternatively, pour the contents into a 40 mL vial and load onto the PTA 30 W/S, which will automatically add IS/SS Standards.

15.2.2 Soil/Sludge/Waste Samples: Prepare a 1g sample (13.2.1.1) if the expected concentration will be within the linear range. If a larger dilution is needed prepare as in 13.2.2 or per EPA method 5035 as specified in TriMatrix SOP GR-04-105.

15.3 Qualitative Analysis:

15.3.1 The qualitative identification of compounds is based on retention time and comparison of sample mass spectra with standard mass spectra. The characteristic ions from the standard mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions are present.

15.3.2 All detected compounds must have a relative retention time (RRT) within ± 0.06 RRT units of the RRT of the standard component.

15.3.3 The relative intensities of the characteristics ions in the sample spectrum must agree within 30% of the relative intensities of these ions in the reference standard spectrum.

15.3.4 When there are resolution problems between compounds, mass spectral results will not give a true spectrum of either compound. When this occurs, examination of the extracted ion current profiles and possibly background subtraction, should be used to help identify whether or not the analytes are present.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Aqueous Samples

16.1.1 Concentration of analyte is determined as follows:

$$\frac{A_x * C_{is}}{A_{is} * RF} * DF = ug/L$$

where:

Ax = Area of the characteristic ion for the target compound.
Ais = Area of the characteristic ion for the specific internal standard.
Cis = Concentration of the specific internal standard in ug/L.
RF = Average Response Factor.
DF = Dilution Factor.

16.1.2 Surrogate Recovery

$$\% \text{ Recovery} = \frac{Ax * Cis * DF * 100}{Ais * RF * TV}$$

TV = true value (40 for aqueous samples)

16.1.3 Matrix Spike Recovery

$$\% \text{ Recovery} = \frac{CS - CC}{TV} \times 100$$

CS = Concentration of sample plus spike (ug/L) as calculated in 16.1.1
CC = Concentration of sample (ug/L) as calculated in 16.1.1
TV = True value (40 for aqueous samples)

16.1.4 % Relative Percent Difference of MS/MSD

$$\% \text{ RPD} = \frac{Cms - Cmsd}{(Cms + Cmsd)/2} x100$$

Cms = Concentration of matrix spike as calculated in 16.1.1.
Cmsd = Concentration of matrix spike duplicate as calculated in 16.1.1.

16.2 Soil/Sludge/Waste (low concentration method)

16.2.1 Concentration of analyte is determined as follows:

$$\frac{mg}{kg} = \frac{Ax * Cis}{Ais * RF} * \frac{5ml}{W * \%S} * \frac{L}{1000ml} * \frac{mg}{1000ug} * \frac{1000g}{kg}$$

where:

Ax = Area of the characteristic ion for the target compound.
Ais = Area of the characteristic ion for the specific internal standard.
Cis = Concentration of the specific internal standard in ug/L.
RF = Average Response Factor
DF = Dilution Factor.
W = wet weight of sample (g)
% S = % solids in decimal form (i.e., 0.90 = 90% solid). Used to calculate dry weight results for soils and sludges only. See Modified EPA Method 160.3.

16.2.2 Surrogate Recovery

16.2.2.1 Calculate as in 16.1.2 if surrogate was added to the purge device or to syringe. (As prepped in section 13.2.1.)

16.2.3 Matrix Spike Recovery

$$\% \text{ Recovery} = \frac{CS - CC}{TV} \times 100$$

Where:

CS = concentration of sample plus spike (mg/kg) as calculated in 16.2.1.
CC = concentration of sample (mg/kg) as calculated in 16.2.1.
TV = true spike value corrected for % solids (generally 0.040 for solid samples prior to % solids correction.)

16.2.4 % Relative Percent Difference of MS/MSD

$$\% \text{ RPD} = \frac{Cms - Cmsd}{(Cms + Cmsd)/2} x100$$

Cms = Concentration of matrix spike as calculated in 16.2.1.
Cmsd = Concentration of matrix spike duplicate as calculated in 16.2.1.

16.3 Soil/Sludge and Waste (High Concentration Method):

16.3.1 Concentration of analyte is determined as follows:

$$\text{mg/kg} = \frac{Ax * C_{IS}}{A_{IS} * RF} * \frac{DF}{\%S} * \frac{V}{W} * \frac{L}{1000ml} * \frac{mg}{1000ug} * \frac{1000g}{kg}$$

Ax = area of the characteristic ion for the target compound
A_{IS} = area of the characteristic ion for the specific internal standard
C_{IS} = concentration of the specific internal standard in ug/L
RF = Average Response Factor
DF = dilution factor
V = volume of solvent added to sample during extraction or dilution (mL)
W = wet weight of sample extracted or diluted (g)
% S = % solids in decimal form (i.e., 0.90 = 90% solid). Used to calculate dry weight results for soils and sludges only. Wastes are calculated on a wet weight basis. See Modified EPA Method 160.3.

16.3.2 Surrogate Recovery

$$\%\ \text{Recovery} = \frac{A_x * C_{IS} * DF * 100}{A_{IS} * RF * TV}$$

TV = true value of surrogate spike

16.3.3 Matrix Spike Recovery

$$\%\ \text{Recovery} = \frac{CS - CC}{TV} \times 100$$

CS = concentration of sample plus spike (mg/kg) as calculated in 16.3.1.
CC = concentration of sample (mg/kg) as calculated in 16.3.1.
TV = true spike value corrected for % solids.

16.3.4 % Relative Percent Difference of MS/MSD

$$\% \text{ RPD} = \frac{Cms - Cmsd}{(Cms + Cmsd)/2} x100$$

Cms = Concentration of matrix spike as calculated in 16.3.1.
Cmsd = Concentration of matrix spike duplicate as calculated in 16.3.1.

16.4 Where applicable, use the regression line fitted to the initial calibration to determine the concentration of analytes.

16.5 Tentatively Identified Compounds (TIC's)
When requested, an estimate of concentration for analytes not present in the quantitation standard(s) will be made. The most recent release of the NIST/EPA/MSDC mass spectral library shall be used to identify TIC's. Compounds with responses less than 10% of the internal standard will not be reported. Relative intensities of the major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum. The relative intensities of the major ions should agree within +/-20%. (Example: For an ion with an abundance of 50% of the standard spectra, the corresponding sample ion abundance must be between 30 and 70%.) MolecuLar ions present in the reference spectrum should be present in the sample spectrum. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting compounds. The formulas given in 16.1.1, 16.2.1, and 16.3.1 will be used to quantitate TIC's with the following modifications: The areas Ax and Ais should be from the total ion chromatogram, the internal standard used will be the closest in elution time to the unknown in the sample that is free of interferences, and the RF used will be 1. The concentration obtained will be reported indicating that the value is estimated. If no valid identification can be made then the compound should be reported as unknown aromatic, unknown hydrocarbon, etc.

17.0 DATA REPORTING AND DELIVERABLES

17.1 The analyst running a set of samples is responsible not only for the quality of the data results themselves, but also for filling out the correct documentation. It is important to document the analyses by correctly filling out, turning in, and filing the correct paperwork. This is required for quality control purposes, and in order to provide the client with defensible data.

17.2 LIMS Reporting

17.2.1 When the analyst has finished running a set of samples the data must be turned in to LIMS. The benchsheets must be filled in completely to insure that the results are reported correctly,

and the data is associated with the right quality control batch. It is important that the quality control batch number from the extraction summary, and the analytical batch information from the 24 hour shift are both filled in correctly. The dilution factor needs to be added if necessary so that the detection limits are raised accordingly. All positive hits should be added by crossing out the detection limit and writing down the final result that goes to the client on the right hand side of the detection limit. This is the result that has already been through all necessary calculations, including the dilution factor. If a dilution was made to the sample and there was a positive hit, the analyst also must include the new elevated detection limit for the analyte with the positive hit by writing it down to the left of the normal detection limit. Surrogates results are reported in amounts found in the extract, amount spiked in the extract, and percent recoveries.

17.2.2 If there are spikes, the quality control benchsheets must also be turned in. In addition to the requirements listed in Section 17.2.1, quality control benchsheets need the spiked amounts, percent recoveries, and percent differences where applicable. If out of control results are present due to extraction or severe matrix problems, the exclude (EXC) box should be checked to prevent the data from biasing the recovery window statistics.

17.2.3 A method blank (MPB) and a Laboratory Fortified Blank (LFB) must be turned in for every analytical batch. If running both 8021 and 601/602 samples, blank benchsheets must be turned in for each method. It is important to remember that a LFB cannot be turned in without first having turned in the associated MPB. A three digit extension (shift and date of the month - i.e. 115 for first shift on the 15th) will be assigned to each MPB and LFB, and this same extension will be added to all associated samples analyzed at the same time.

17.2.4 If internal chain-of-custody is required it is very important that the COC sheet is filled out correctly. All days that the analyst is in possession of the samples need to be accounted for.

17.2.5 All LIMS benchsheets (including the COC sheets) are to be placed in the correctly colored folders and turned into the data entry person responsible for entering the results. The blue folders containing the MPB's also should have the blank raw data quantitation reports and chromatograms included. All rush or past due projects should be in red or green folders.

17.3 Laboratory Required Paperwork

17.3.1 All run, maintenance, tape, and standard logbooks must be filled in completely and correctly. Corrections are to be made with a single lineout and then initialed and dated, not a writeover, and blank lines in the run logbook should be Z'd out, initialed and dated.

17.3.2 All ICV and CCV standard runs must be archived in their correct binders.

17.3.3 All LIMS documentation (except for the sample and quality control benchsheets), and the raw data (except for the blanks and what is listed in 17.3.2) should be placed in the correct folder and turned into the Volatile Laboratory technician who will record the date, time, and contents that were turned in.

17.4 Rounding and Significant Figures

17.4.1 Rounding is performed on the final quantitated result only. All non-quality control results are rounded to 2 significant figures.

17.4.2 Quality control results are rounded to 3 significant figures.

17.4.3 Percent recoveries and percent differences are reported as whole numbers up to 3 significant figures.

18.0 QUALITY ASSURANCE

18.1 BFB (Section 14.1)

18.2 5 Point Curve (Section 14.2)

18.3 Continuing Calibration Standard (Section 14.3)

18.4 Blank (Section 14.4)

18.5 Method Preparation Blank (MPB)

An MPB must be analyzed each day prior to running samples, and must have no positive results above the laboratory reporting limits for any analyte. If this requirement is not met, sample analysis may not begin, and corrective action must be performed. The only exception to this is for common laboratory contaminants (i.e. methylene chloride, acetone, and MEK), which may contain up to five times the laboratory reporting limit. However, any positive results reported for those analytes with concentrations between one and five times that reported in the blank will be qualified as estimated. If the samples to be analyzed are from methanol preparation, the MPB must contain 100 ul of methanol. If samples containing the bisulfate preservative are being analyzed, a MPB containing sodium bisulfate must also be analyzed.

18.6 Laboratory Fortified Blank (LFB)

A LFB is required with each 12 hour analytical batch, or every 20 samples, whichever is more frequent. It is taken from the results of the CCV at a mid-level concentration. The LFB serves as a check of method/extraction performance in the event that matrix spike recoveries are not within applicable control limits. Recoveries must be calculated and compared to LIMS control limits. Analysis must be stopped and the problem corrected if recoveries are outside the control limits. For aqueous and low level soil samples, the LFB may be either from the same source or separate source as the curve. For high level soils and wastes, see section 13.2.2.4 or TriMatrix SOP GR-04-105 (Method 5035) for LFB preparation procedures. Any sample analyzed in a batch with a failing LFB must be re-analyzed for the failing parameters. If this is not possible, all data must be qualified as estimated.

18.7 Matrix Spikes (MS, MSD)
Duplicate matrix spikes at a mid-level concentration are analyzed for each batch of 20 samples of the same matrix. Recoveries and % RSD are compared to statistically derived LIMS control limits. Recoveries/% RSD outside these limits must be qualified. (See TriMatrix Volatile Laboratory Corrective Actions SOP - GR-03-124.)

18.8 Spike Reporting

18.8.1 All routinely requested 8260B analytes listed in standards 1A & 1B will be added when spiking. All of these analytes will be reported at least twice each month for each matrix in order to generate quality control limits for all analytes.

18.8.2 For both LFB and matrix spikes, a list of 5 compounds is reported as follows for method 8260B. (This list is subject to client requests).

1. 1,1-Dichloroethylene
2. Trichloroethylene
3. Chlorobenzene
4. Benzene
5. Toluene

18.8.3 **For all State of Wisconsin samples, all of the spiked analytes will be reported when analyzing both LFB's and matrix spikes.**

18.9 Laboratory Control Sample (LCS)
After a new calibration curve is generated a separate source LCS must be analyzed for all target compounds. The windows are ± 25%. Analysis must be stopped and the problem corrected if recoveries are outside these windows.

18.10 Internal Standard Responses

18.10.1 The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. The retention time for all internal standards must be within ± 30 sec from the last continuing calibration check standard. The EICP area for all internal standards must stay within a factor of two (-50% to +100%) from the last daily calibration standard check.

18.10.2 The EICP area for all internal standards in samples must stay within a factor of two (-50% to +100%) from the last daily calibration standard check.

18.10.3 If it appears that a sample matrix effect is causing a deviation, the sample will not be re-run. If there is no apparent reason for the deviation, then the sample will be re-run. If it works the second time, the second run will be reported. If it does not work this second time, a dilution of the sample will be performed until the internal standard meets criteria. Detection limits will be raised accordingly. If a dilution still does not meet criteria then all compounds associated with the internal standard that was out will be considered estimated. If many samples are out of control for no apparent reason, the mass spectrometer needs to be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning are necessary.

18.11 Surrogate Recoveries

18.11.1 All surrogates must fall within statistically derived LIMS control limits or method windows if LIMS windows are wider. If any surrogate compound is out of control due to obvious matrix problems, the sample will not be re-run and the sample will be qualified as estimated. If the sample has a surrogate that is out for no apparent reason, the sample will be re-run. If the re-run works, the first run will be discarded, and the second run reported. If many samples are out of control for no apparent reason, the mass spectrometer needs to be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning are necessary.

18.11.2 If a high level sample is solvent extracted or diluted (13.2.2) and the true value of the surrogate is less than 8 ug/L, surrogate recoveries will not be reported.

18.12 Method 624 Modifications

This SOP was written primarily as method 8260B, however the following modifications are allowed for method 624.

18.12.1 The 12 hour shift is replaced by a 24 hour shift (14.1.2). BFB and a continuing calibration (14.3.1) standard are still required, only they are run every 24 hours instead of every 12.

18.12.2 A 3 point curve can be used in place of a 5 point curve (14.2.1).

18.12.3 SPCC's and CCC's are not used (14.2.5 and 14.2.6). Instead the RF's for every compound *listed on the Method 624 list must have ≤35 % RSD for the curve to be valid.*

18.12.4 The continuing calibration standard also does not use SPCC's and CCC's (14.3.1.1 and 14.3.1.2). The RF for every compound in the 40 ug/L continuing calibration standard is compared with the corresponding calibration acceptance criteria found in Table 6. If the responses for all parameters of interest fall within the designated ranges, analysis of actual samples can begin. If any individual RF falls outside the range, a new continuing calibration standard, or a new curve will be run.

18.12.5 There is no criteria for internal standard areas or retention times in Method 624. The Method 8260B criteria will be followed, but are not mandatory (18.10).

18.12.6 Every compound of interest that is on the Method 624 list must be in the matrix spikes, laboratory control samples, and LFB's, not just the limited list from 18.8.

18.12.7 Acrolein and Acrylonitrile may only be screened by GC/MS. All positive results must therefore be considered estimated.

19.0 ANALYST CERTIFICATION/METHOD VALIDATION

19.1 Before the analysis of any actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running a one time analyst certification. While the analyst certification is not instrument dependent, this certification is required on every instrument that will be running samples to demonstrate instrument ability to generate acceptable accuracy and precision.

19.2 Prepare a quality control check sample spiking standard at a level which will give concentrations at 20 ug/L per compound.

19.3 Analyze the four 1 ml samples following the SOP.

19.4 Calculate the average recovery (x) in ug/L, and the standard deviation of the recovery (s) in ug/L, for each analyte using the four results.

19.4.1 For each analyte x must be in the range 70-130% and s must be less than or equal to 20. If s and x for all analytes meet the acceptance criteria, the analyst certification is good. The analyst and the system are now authorized to run samples by this method. If any individual s

exceeds the precision limit or any individual falls outside the range for accuracy, then the system performance is unacceptable for that analyte.

19.4.2 When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst must proceed according to 19.5.

19.5 Locate and correct the source of the problem and repeat the test for all analytes that failed to meet the criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds beginning with Section 19.1. Samples may not be analyzed by any analyst or on any instrument until the analyst certification has been successfully completed. Copies of the successful analyst certifications/method validations spreadsheet and raw data should be given to the Quality Assurance Manager.

19.6 Method Detection Limit Studies

19.6.1 A Method Detection Limit (MDL) study must be performed for each analyte, for both waters and soils, to be quantitated by method 8260B1. All GC/MS instruments must perform MDL studies for each compound. Results obtained for analytes for which an MDL study has not been performed must be considered estimated. The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the value is above zero. Actual Reporting Limits (RLs)are derived from the MDL study. RLs are the amount spiked for the MDL study provided that the MDL passed. The RL actually achieved in any given analysis will vary depending on instrument sensitivity, matrix effects, and dilutions.

19.6.2 The procedure followed for a MDL study is based on the method given in the Code of Federal Regulations, Part 136, Appendix B, 7-1-97, revision 1.11. All Method 8260B quality control procedures governing the analyses must be followed.

19.6.3 At least seven replicate analyses are performed using reagent water or sand, spiked with all analytes of interest, at the estimated RL concentration.

19.6.4 If the amount spiked is ≥1 and ≤5 times the calculated MDL, and there are no 0 percent recoveries in the set of seven, the MDL result is acceptable. If not the MDL must be re-run, only for the compounds that did not pass. If the study needs to be repeated at a different concentration, an entire set of seven needs to be re-run. If the study does not pass due to poor reproducibility on one of the samples, only that run needs to be re-analyzed.

20.0 REFERENCES

Approved By: _____ 5/28/98 QA Manager

Approved By: _____ 5/28/98 Area Manager

20.1 USEPA, SW846, Update III, Revision 2, 3rd Edition, Method 8260B, December 1996.

20.2 USEPA 40 CFR Method 624, Pt. 136, App. A., 1997.

20.3 USEPA CLP - Statement of Work, August 1991 Revision, Section IV.

21.0 ATTACHMENTS/APPENDICES

TABLE 1A
COMPOUND LIST, CAS#, AND REPORTING LIMITS FOR STANDARD I

Compound	CAS#	Aqueous DL (ug/L)	Soil DL (mg/kg)
Acetone	67-64-1	50	0.10
Acrolein	107-02-8	5.0	0.010
Acrylonitrile	107-13-1	1.0	0.010
Benzene	71-43-2	1.0	0.010
Bromobenzene	108-86-1	1.0	0.010
Bromochloromethane	74-97-5	1.0	0.010
Bromodichloromethane	75-27-4	1.0	0.010
Bromoform	75-25-2	1.0	0.010
Bromomethane	74-83-9	1.0	0.010
Carbon DisuLfide	75-15-0	5.0	0.10
Carbon Tetrachloride	56-23-5	1.0	0.010
Chlorobenzene	108-90-7	1.0	0.010
Chloroethane	75-00-3	1.0	0.010
2-Chloroethyl Vinyl Ether	110-75-8	10	0.10
Chloroform	67-66-3	1.0	0.010
Chloromethane	74-87-3	1.0	0.010
2-Chlorotoluene	95-49-8	1.0	0.010
4-Chlorotoluene	106-43-4	1.0	0.010
cis-1,2-Dichloroethylene	156-59-2	1.0	0.010
cis-1,3-Dichloropropylene	10061-01-5	1.0	0.010
Dibromochloromethane	124-48-1	1.0	0.010
1,2-Dibromo-3-Chloropropane	96-12-8	1.0	0.010
1,2-Dibromoethane	106-93-4	1.0	0.010

Approved By: _____ 5/28/98 (QA Manager) Approved By: _____ 5/28/98 (Area Manager)

TABLE 1A
COMPOUND LIST, CAS#, AND REPORTING LIMITS FOR STANDARD I
(Continued)

Compound	CAS#	Aqueous DL (ug/L)	Soil DL (mg/kg)
Dibromomethane	74-95-3	1.0	0.010
1,2-Dichlorobenzene	95-50-1	1.0	0.010
1,3-Dichlorobenzene	541-73-1	1.0	0.010
1,4-Dichlorobenzene	106-46-7	1.0	0.010
Dichlorodifluoromethane	75-71-8	1.0	0.010
1,1-Dichloroethane	75-34-3	1.0	0.010
1,2-Dichloroethane	107-06-2	1.0	0.010
1,1-Dichloroethylene	75-35-4	1.0	0.010
Dichlorofluoromethane	73-43-4	1.0	0.010
1,2-Dichloropropane	78-87-5	1.0	0.010
1,3-Dichloropropane	142-28-9	1.0	0.010
2,2-Dichloropropane	594-20-7	1.0	0.010
1,1-Dichloropropylene	563-58-6	1.0	0.010
Ethyl Ether	60-29-7	10	0.10
Ethylbenzene	100-41-4	1.0	0.010
Heptane	142-82-5	10	0.10
Hexachlorobutadiene	87-68-3	5.0	0.050
2-Hexanone	591-78-6	50	0.10
Iodomethane	74-88-4	1.0	0.010
Isobutyl acetate	110-19-0	10	0.10
Isopropanol	67-63-0	50	0.40

TABLE 1A
COMPOUND LIST, CAS#, AND REPORTING LIMITS FOR STANDARD I
(Continued)

Compound	CAS#	Aqueous DL (ug/L)	Soil DL (mg/kg)
Isopropylbenzene	98-82-8	1.0	0.010
Methyl Ethyl Ketone (2-Butanone)	78-93-3	50	0.10
4-Methyl-2-Pentanone (MIBK)	108-10-1	50	0.10
Methyl tert-Butyl Ether (MTBE)	1634-04-4	50	0.10
Methylene Chloride	75-09-2	1.0	0.010
n-Butyl acetate	123-86-4	10	0.020
n-Butylbenzene	104-51-8	1.0	0.010
n-Propylbenzene	103-65-1	1.0	0.010
Naphthalene	91-20-3	5.0	0.050
p-Isopropyltoluene	99-87-6	1.0	0.010
sec-Butylbenzene	135-98-8	1.0	0.010
Styrene	100-42-5	1.0	0.010
tert-Butylbenzene	98-06-6	1.0	0.010
1,1,1,2-Tetrachloroethane	630-20-6	1.0	0.010
1,1,2,2-Tetrachloroethane	79-34-5	1.0	0.010
Tetrachloroethylene	127-18-4	1.0	0.010
Toluene	108-88-3	1.0	0.010
Trans-1,2-Dichloroethylene	156-60-5	1.0	0.010
Trans-1,3-Dichloropropylene	10061-02-6	1.0	0.010
1,2,3-Trichlorobenzene	87-61-6	1.0	0.010
1,2,4-Trichlorobenzene	120-82-1	1.0	0.010

TABLE 1A
COMPOUND LIST, CAS#, AND REPORTING LIMITS FOR STANDARD I
(Continued)

Compound	CAS#	Aqueous DL (ug/L)	Soil DL (mg/kg)
1,1,1-Trichloroethane	71-55-6	1.0	0.010
1,1,2-Trichloroethane	79-00-5	1.0	0.010
Trichloroethylene	79-01-6	1.0	0.010
Trichlorofluoromethane	75-69-4	1.0	0.010
1,2,3-Trichloropropane	96-18-4	1.0	0.010
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	1.0	0.010
1,2,4-Trimethylbenzene	95-63-6	1.0	0.010
1,3,5-Trimethylbenzene	108-67-8	1.0	0.010
Vinyl Acetate	108-05-4	5.0	0.10
Vinyl Chloride	75-01-4	1.0	0.010
m,p-Xylene	999-99-9*	2.0	0.020
o-Xylene	95-47-6	1.0	0.010
Xylene, Total	1330-20-7	3.0	0.030
trans-1,4-Dichloro-2-butylene	110-57-6	5.0	0.050

*m-xylene 108-38-3
*p-xylene 106-42-3

Approved By: _____ 5/28/98 (QA Manager) Approved By: _____ 5/28/98 (Area Manager)

TABLE 1B
COMPOUND LIST, CAS #, AND REPORTING LIMITS FOR STANDARD II

Compound	CAS#	Aqueous DL (ug/L)	Soil DL (mg/kg)
Acetonitrile	75-05-8	10	0.10
2-Butanol	15892-23-6	50	0.50
n-Butanol	71-36-3	40	0.23
t-Butanol	75-65-0	50	0.50
Cyclohexane	110-82-7	10	0.10
Cyclohexanone	108-94-1	50	0.10
2,3-Dichloro-1-propylene	78-88-6	1.0	0.010
1,2-Diethylbenzene	135-01-3	5.0	0.050
1,3-Diethylbenzene	141-93-5	5.0	0.050
1,4-Diethylbenzene	105-05-5	5.0	0.050
Dimethyl disuLfide	624-92-0	5.0	0.050
1,4-Dioxane	123-91-1	300	3.0
Ethanol	64-17-5	100	1.0
Ethyl Acetate	141-78-6	50	0.50
Hexachloroethane	67-72-1	5.0	0.050
Hexane	110-54-3	10	0.10
Isobutanol	78-83-1	50	0.50
Isopropyl ether	108-20-3	5.0	0.050
Methacrylonitrile	126-98-7	50	0.50
Methylcyclohexane	108-87-2	10	0.10
Methylcyclopentane	96-37-7	10	0.10
2-Nitropropane	79-46-9	10	0.10
n-Propanol	71-23-8	100	1.0
Propionitrile	107-12-0	50	0.50
trans-1,4-Dichloro-2-butylene	110-57-6	5.0	0.050

Approved By: _____ 5/29/98 _____ Approved By: _____ 5/27/98 _____
QA Manager Area Manager

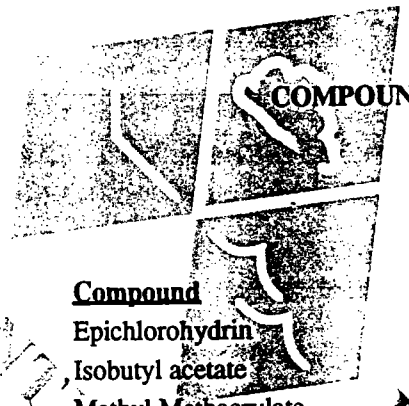


TABLE 1B
COMPOUND LIST, CAS #, AND REPORTING LIMITS FOR STANDARD II (continued)

Compound	CAS#	Aqueous DL (ug/L)	Soil DL (mg/kg)
Epichlorohydrin	106-89-8	5.0	0.010
Isobutyl acetate	110-19-0	10	0.10
Methyl Methacrylate	80-62-6	100	1.0
Tetrahydrofuran	109-99-9	10	0.10
n-Butyl acetate	123-86-4	10	0.020
Allyl Chloride	107-05-1	5.0	0.050

Approved By: _____ 5/28/98
QA Manager

Approved By: _____ 5/25/98
Area Manager

TABLE 2A
8260 STANDARD I COMPOUNDS AND CONCENTRATIONS

STD	Compound	Conc. (ug/mL)
IE	Acetone	100
IC	Acrolein**	100
IC	Acrylonitrile**	100
IA	Benzene	100
IA	Bromobenzene	100
IA	Bromochloromethane	100
IA	Bromodichloromethane	100
IA	Bromoform	100
IB	Bromomethane*	100
IE	Carbon DisuLfide	100
IA	Carbon Tetrachloride	100
IA	Chlorobenzene	100
IB	Chloroethane*	100
ID	2-Chloroethyl Vinyl Ether	100
IA	Chloroform	100

Approved By: RK 5/28/98 QA Manager Approved By: DB 5/27/98 Area Manager

TABLE 2A
8260 STANDARD I COMPOUNDS AND CONCENTRATIONS
(Continued)

STD	Compound	Conc. (ug/mL)
IB	Chloromethane*	100
IA	2-Chlorotoluene	100
IA	4-Chlorotoluene	100
IA	cis-1,2-Dichloroethylene	100
IA	cis-1,3-Dichloropropylene	100
IA	Dibromochloromethane	100
IA	1,2-Dibromo-3-Chloropropane	100
IA	1,2-Dibromoethane	100
IA	Dibromomethane	100
IA	1,2-Dichlorobenzene	100
IA	1,3-Dichlorobenzene	100
IA	1,4-Dichlorobenzene	100
IB	Dichlorodifluoromethane*	100
IA	1,1-Dichloroethane	100
IA	1,2-Dichloroethane	100
IA	1,1-Dichloroethylene	100
IA	Dichlorofluoromethane*	100
IA	1,2-Dichloropropane	100
IA	1,3-Dichloropropane	100
IA	2,2-Dichloropropane	100
IA	1,1-Dichloropropylene	100
IE	Ethyl Ether	100

TABLE 2A
8260 STANDARD I COMPOUNDS AND CONCENTRATIONS
(Continued)

STD	Compound	Conc. (ug/mL)
IA	Ethylbenzene	100
IE	Heptane	100
IA	Hexachlorobutadiene	100
IE	2-Hexanone	100
IE	Iodomethane	100
	Isobutyl acetate	100
IE	Isopropanol	100
IA	Isopropylbenzene	100
IE	Methyl Ethyl Ketone (2-Butanone)	100
	Methyl Methacrylate	100
IE	4-Methyl-2-Pentanone (MIBK)	100
IE	Methyl tert-Butyl Ether (MTBE)	100
IA	Methylene Chloride	100
	n-Butyl acetate	100
IA	n-Butylbenzene	100
IA	n-Propylbenzene	100
IA	Naphthalene	100
IA	p-Isopropyltoluene	100
IA	sec-Butylbenzene	100

TABLE 2A
8260 STANDARD I COMPOUNDS AND CONCENTRATIONS
(Continued)

STD	Compound	Conc. (ug/mL)
IA	Styrene	100
IA	tert-Butylbenzene	100
IA	1,1,1,2-Tetrachloroethane	100
IA	1,1,2,2-Tetrachloroethane	100
IA	Tetrachloroethylene	100
IA	Toluene	100
IA	Trans-1,2-Dichloroethylene	100
IA	Trans-1,3-Dichloropropylene	100
IA	1,2,3-Trichlorobenzene	100
IA	1,2,4-Trichlorobenzene	100
IA	1,1,1-Trichloroethane	100
IA	1,1,2-Trichloroethane	100
IA	Trichloroethylene	100
IB	Trichlorofluoromethane	100
IA	1,2,3-Trichloropropane	100
IE	1,1,2-Trichloro-1,2,2-trifluoroethane	100
IA	1,2,4-Trimethylbenzene	100
IA	1,3,5-Trimethylbenzene	100
IE	Vinyl Acetate	100
IB	Vinyl Chloride*	100
IA	m,p-Xylene	200
IA	o-Xylene	100
IA	Xylene, Total	300

TABLE 2B
8260 APPENDIX IX COMPOUNDS AND CONCENTRATIONS

Compound	Conc. (ug/mL)
Acetonitrile	250
sec-Butanol	250
1-Butanol	250
t-Butanol	250
Allyl Chloride	250
Cyclohexanone	250
2,3-Dichloro-1-propene	250
Epichlorohydrin	250
p-Dioxane	250
Ethanol	250
Ethyl Acetate	250
Hexachloroethane	250
n-Hexane	250
Isobutyl Alcohol	250
Isopropyl Ether	250
Methacrylonitrile	250
2-Nitropropane	250
Propionitrile	250
1-Propanol	250
Tetrahydrofuran	250

TABLE 5A
ELUTION ORDER, QUANTITATION AND CHARACTERISTIC IONS, INTERNAL STANDARDS, AND SURROGATES FOR STANDARD I

Compound	Quantitation Ion	Secondary Ion(s)	Internal Standard
Dichlodifluoromethane	85	87, 50	1
Chloromethane	50	52, 49	1
Vinyl Chloride	62	64, 47	1
Bromomethane	94	96, 93	1
Chloroethane	45	43	1
Trichlorofluoromethane	101	103, 105	1
Dichlorofluoromethane	67	69, 47	1
Ethyl Ether	45	59, 73	1
Acrolein	55	56, 53	1
1,1-Dichloroethylene	96	61, 98	1
1,1,2-Trichloro-1,2,2-trifluoroethane	101	103, 151	1
Iodomethane	142	127, 141	1
Carbon DisuLfide	76	78, 77	1
Acetone	43	58, 42	1
Allyl Chloride	76	39, 41	1
Isopropanol	45	59, 43	1
Methylene Chloride	49	51, 84	1
Acrylonitrile	52	54, 53	1
Trans-1,2-Dichloroethylene	96	61, 98	1

Approved By: _____ 5/28/98 (QA Manager) Approved By: _____ 5/28/98 (Area Manager)

TABLE 5A
ELUTION ORDER, QUANTITATION AND CHARACTERISTIC IONS, INTERNAL STANDARDS, AND SURROGATES FOR STANDARD I
(Continued)

Compound	Quantitation Ion	Secondary Ion(s)	Internal Standard
Methyl tert-Butyl Ether (MTBE)	73	43, 57	1
1,1-Dichloroethane	63	65,83	1
Vinyl Acetate	43	42, 44	1
2,2-Dichloropropane	77	41, 79	1
cis-1,2-Dichloroethylene	96	61, 63	1
Methyl Ethyl Ketone (2-Butanone)	43	72, 57	1
Bromochloromethane	49	130, 128	1
Tetrahydrofuran	71	41, 42	1
Chloroform	83	85, 47	1
1,1,1-Trichloroethane	97	99, 61	1
SUR:Dibromofluoromethane	113	111, 192	1
IS:Pentafluorobenzene	137	168, 99	1
Carbon Tetrachloride	117	119, 121	2
1,1-Dichloropropylene	75	110, 77	2
Benzene	78	51, 50	2
1,2-Dichloroethane	62	64, 49	2
Heptane	41	57, 71	2
IS:1,4-Difluorobenzene	114	88, 63	2
Trichloroethylene	130	95, 132	2
1,2-Dichloropropane	63	62, 76	2
Dibromomethane	93	95, 174	2
Methyl Methacrylate	41	69, 100	2
Bromodichloromethane	83	85, 129	2
2-Chloroethyl Vinyl Ether	63	65, 106	2

Approved By: _____ 5/28/98 Approved By: _____ 5/28/98
QA Manager Area Manager

TABLE 5A
ELUTION ORDER, QUANTITATION AND CHARACTERISTIC IONS, INTERNAL STANDARDS, AND SURROGATES FOR STANDARD I
(Continued)

Compound	Quantitation Ion	Secondary Ion(s)	Internal Standard
Epichlorohydrin	49	57, 62	2
cis-1,3-Dichloropropylene	75	77, 110	2
4-Methyl-2-Pentanone (MIBK)	43	58, 85	2
SUR:d8-Toluene	98	100,70	2
Toluene	91	92, 65	2
n-Butyl acetate	43	56, 57	2
Trans-1,3-Dichloropropylene	75	77, 110	2
Ethyl methacrylate	41	99, 69	2
1,1,2-Trichloroethane	97	83, 85	2
Tetrachloroethylene	166	129, 168	3
1,3-Dichloropropane	76	78, 41	3
2-Hexanone	43	58, 85	3
Isobutyl acetate	43	41, 56	3
Dibromochloromethane	129	127, 131	3
1,2-Dibromoethane	109	107, 188	3
IS:d5-Chlorobenzene	82	117, 119	3
Chlorobenzene	112	77, 114	3
1,1,1,2-Tetrachloroethane	131	133, 119	3
Ethylbenzene	91	106, 51	3
m,p-Xylene	91	106, 51	3
o-Xylene	91	106, 51	3
Styrene	104	78, 51	3
Bromoform	173	175, 79	3
Isopropylbenzene	105	120, 79	4

Approved By: _____ 5/28/98 (QA Manager)

Approved By: _____ 5/28/98 (Area Manager)

TABLE 5A
ELUTION ORDER, QUANTITATION AND CHARACTERISTIC IONS, INTERNAL STANDARDS, AND SURROGATES FOR STANDARD I
(Continued)

Compound	Quantitation Ion	Secondary Ion(s)	Internal Standard
SUR:4-Bromofluorobenzene	95	174, 176	3
Bromobenzene	77	156, 158	4
1,1,2,2-Tetrachloroethane	83	85, 95	4
1,2,3-Trichloropropane	75	49, 110	4
n-Propylbenzene	91	120, 65	4
2-Chlorotoluene	126	91, 63	4
1,3,5-Trimethylbenzene	105	120, 77	4
4-Chlorotoluene	91	126, 63	4
tert-Butylbenzene	119	91, 134	4
1,2,4-Trimethylbenzene	105	120, 77	4
sec-Butylbenzene	105	134, 91	4
1,3-Dichlorobenzene	146	111, 148	4
p-Isopropyltoluene	119	134, 91	4
IS:d4-1,4-Dichlorobenzene	152	150	4
1,4-Dichlorobenzene	146	111, 148	4
1,2-Dichlorobenzene	146	111, 148	4
n-Butylbenzene	91	92, 134	4
1,2-Dibromo-3-Chloropropane	75	155, 157	4
1,2,4-Trichlorobenzene	180	182, 145	4
Hexachlorobutadiene	225	227, 260	4
Naphthalene	128	102, 51	4
1,2,3-Trichlorobenzene	180	182, 145	4

Approved By: RD 5/28/98 QA Manager

Approved By: JS 5/28/98 Area Manager

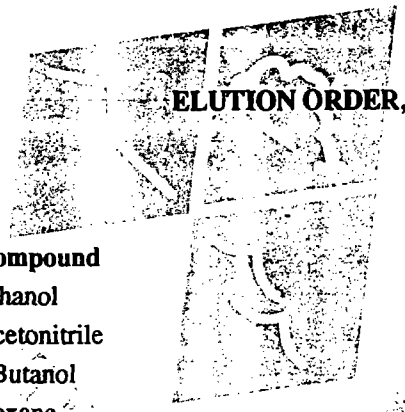


TABLE 5B
ELUTION ORDER, QUANTITATION AND CHARACTERISTIC IONS, FOR STANDARD II

Compound	Quantitation Ion	Secondary Ion(s)	Internal Standard
Ethanol	45	43	1
Acetonitrile	41	40, 54	1
t-Butanol	59	41, 43	1
Hexane	41	56, 57	1
Isopropylether	45	69, 86	1
n-Propanol	42	59, 41	1
Methylcyclopentane	56	41, 69	1
Propionitrile	54	52, 56	1
Ethyl Acetate	43	61, 45	1
Methacrylonitrile	67	41, 52	1
2-Butanol	45	59, 57	1
Sur: Dibromofluoromethane	113	111, 79	1
Cyclohexane	41	39, 57	1
IS: Pentafluorobenzene	137	168, 99	1
Isobutanol	41	43, 42	2
IS: 1,4-Difluorobenzene	114	88, 63	2
n-Butanol	41	39, 56	2
Methylcyclohexane	55	83, 98	2
2,3-Dichloro-1-propylene	75	77, 110	2
1,4-Dioxane	88	57, 43	2
2-Nitropropane	41	43, 46	2
Hexachloroethane	117	119, 201	2
Dimethyl disuLfide	45	79, 94	2
Sur: d-8-Toluene	98	100, 70	2
IS: d5-Chlorobenzene	82	117, 119	3
Cyclohexanone	55	42, 98	3

Approved By: _____ 5/28/98 Approved By: _____ 5/28/98
QA Manager Area Manager

TABLE 5B
ELUTION ORDER, QUANTITATION AND CHARACTERISTIC IONS, FOR STANDARD II
(Continued)

Compound	Quantitation Ion	Secondary Ion(s)	Internal Standard
Sur: 4-Bromofluorobenzene	95	174, 176	3
trans-1,4-Dichloro-2-butylene	53	75, 89	4
IS: d4-1,4-Dichlorobenzene	152	150	4
1,3-Diethylbenzene	105	119, 134	4
1,4-Diethylbenzene	105	119, 134	4
1,2-Diethylbenzene	105	119, 134	4

Subject: Volatile Organic Compounds in Water by Purge and Trap Capillary Column Gas Chromatography/Mass Spectrometry

USEPA Method 8260A
USEPA Method 624

Procedure No: GR-04-104
Revision No: 3.0
Effective Date: 05/28/98

TABLE 6
CALIBRATION AND QC ACCEPTANCE CRITERIA
METHOD 624

Parameter	Range for Q (ug/L)
Benzene	12.8-27.2
Bromodichloromethane	13.1-26.9
Bromoform	14.2-25.8
Bromomethane	2.8-37.2
Carbon tetrachloride	14.6-25.4
Chlorobenzene	13.2-26.8
Chloroethane	7.6-32.4
2-Chloroethylvinyl ether	D-44.8
Chloroform	13.5-26.5
Chloromethane	D-40.8
Dibromochloromethane	13.5-26.5
1,2-Dichlorobenzene	12.6-27.4
1,3-Dichlorobenzene	14.6-25.4
1,4-Dichlorobenzene	12.6-27.4
1,1-Dichloroethane	14.5-25.5
1,2-Dichloroethane	13.6-26.4
1,1-Dichloroethylene	10.1-29.9
trans-1,2-Dichloroethylene	13.9-26.1
1,2-Dichloropropane	6.8-33.2
cis-1,3-Dichloropropylene	4.8-35.2
trans-1,3-Dichloropropylene	10.0-30.0
Ethyl benzene	11.8-28.2
Methylene chloride	12.1-27.9
1,1,2,2-Tetrachloroethane	12.1-27.9

Approved By: _____ 5/28/98 Approved By: _____ 5/28/98
QA Manager Area Manager

TABLE 6
CALIBRATION AND QC ACCEPTANCE CRITERIA
METHOD 624
(Continued)

Parameter	Range for Q (ug/L)
Tetrachloroethylene	14.7-25.3
Toluene	14.9-25.1
1,1,1-Trichloroethane	15.0-25.0
1,1,2-Trichloroethane	14.2-25.8
Trichloroethylene	13.3-26.7
Trichlorofluoromethane	9.6-30.4
Vinyl chloride	0.8-39.2

D=Detected result must be greater than zero.

Approved By: [signature] 5/28/98 QA Manager

Approved By: [signature] 5/28/98 Area Manager

Response Factor Report

Method : C:\SAT132\QUANT\8260B-3.M (RTE Integrator)
Title : Saturn 132
Last Update : Fri Feb 26 15:11:05 1999
Response via : Initial Calibration

Calibration Files
low =CAP0301.D mlow =CBP0301.D mid =CCP0301.D
mhi =CDP0301.D high =CEP0301.D =

		Compound	low	mlow	mid	mhi	high	Avg	%RSI
1)	I	IS: Flourobenzene	----------	----------	ISTD	----------	----------		
2)	T	Dichlorodifluoromet	0.149	0.172	0.176	0.205	0.197	0.180	12.31
3)	TP	Chloromethane	0.120	0.131	0.134	0.161	0.159	0.141	12.78
4)	TC	Vinyl Chloride	0.195	0.195	0.202	0.229	0.229	0.210	8.45
5)	T	Bromomethane	0.215	0.193	0.208	0.275	0.282	-----	
							QO	A= 0.009 R=0.999	
								B= 0.238	
6)	T	Chloroethane	0.021	0.021	0.022	0.024	0.019	0.021	7.20
7)	T	Trichlorofluorometh	0.471	0.476	0.486	0.571	0.552	0.511	9.20
8)	T	Ethyl Ether	0.128	0.140	0.142	0.163	0.143	0.143	8.88
9)	T	Acrolein	0.019	0.017	0.016	0.019	0.021	0.019	12.29
10)	TCM	1,1-Dichloroethene	0.250	0.259	0.264	0.309	0.291	0.275	8.90
11)	T	Trichlorotrifluoroe	0.367	0.375	0.384	0.441	0.421	0.398	8.07
12)	T	Iodomethane	0.519	0.612	0.661	0.862	0.875	-----	
							LO	M= 0.863 R=0.999	
13)	T	Carbon Disulfide	0.567	0.561	0.569	0.646	0.602	0.589	6.09
14)	T	Acetone	0.177	0.146	0.121	0.124	0.116	-----	
							LO	M= 0.118 R=0.9	
15)	T	Isopropanol	0.012	0.010	0.009	0.009	0.008	-----	
							QO	A= -0.001 R=0.998	
								B= 0.010	
16)	T	Methylene Chloride	0.606	0.597	0.600	0.694	0.684	0.636	7.61
17)	T	Acrylonitrile	0.156	0.122	0.128	0.146	0.142	0.139	9.82
18)	T	trans-1,2-Dichloroe	0.283	0.288	0.296	0.353	0.309	0.306	9.2
19)	T	Methyl(tert)Butyl E	0.825	0.816	0.860	1.024	0.954	0.896	10.0
20)	TP	1,1-Dichloroethane	0.399	0.414	0.433	0.509	0.493	0.450	10.8
21)	T	Vinyl Acetate	0.437	0.445	0.468	0.592	0.572	0.503	14.5
22)	T	2,2-Dichloropropane	0.521	0.463	0.549	0.644	0.667	0.569	15.0
23)	T	cis-1,2-Dichloroeth	0.318	0.320	0.321	0.367	0.376	0.341	8.4
24)	T	Methyl Ethyl Ketone	0.171	0.163	0.149	0.163	0.151	0.160	5.9
25)	T	Bromochloromethane	0.380	0.388	0.389	0.474	0.430	0.412	9.5
26)	TC	Chloroform	0.580	0.605	0.605	0.679	0.660	0.626	6.6
27)	T	1,1,1-Trichloroetha	0.645	0.650	0.674	0.796	0.761	0.705	9.7
28)	S	SUR: Dibromofluorom	0.370	0.346	0.304	0.320	0.304	0.329	8.7
29)	T	Carbon Tetrachlorid	0.481	0.486	0.502	0.569	0.570	0.522	8.5
30)	T	1,1-Dichloropropene	0.457	0.466	0.461	0.527	0.516	0.486	6.8
31)	TM	Benzene	1.236	1.224	1.240	1.466	1.458	1.325	9.4
32)	T	1,2-Dichloroethane	0.500	0.486	0.497	0.582	0.560	0.525	8.1
33)	T	Heptane	0.566	0.574	0.563	0.665	0.657	0.605	8.4
34)	S	SUR: 1,2-Dichloroet	0.393	0.387	0.383	0.390	0.403	0.391	1.9
35)	TM	Trichloroethene	0.328	0.328	0.333	0.384	0.369	0.348	7.4
36)	TC	1,2-Dichloropropane	0.230	0.224	0.223	0.257	0.241	0.235	5.9
37)	T	Dibromomethane	0.256	0.249	0.252	0.296	0.279	0.267	7.6

L = Linear LO = Linear+Origin Q = Quad QO = Quad+Origin R = Corr. Coef
(#) = Out of Range

Response Factor Report

Method : C:\SAT132\QUANT\8260B-3.M (RTE Integrator)
Title : Saturn 132
Last Update : Fri Feb 26 15:11:05 1999
Response via : Initial Calibration

Calibration Files
low =CAP0301.D mlow =CBP0301.D mid =CCP0301.D
mhi =CDP0301.D high =CEP0301.D =

		Compound	low	mlow	mid	mhi	high	Avg	%RSD
38)	T	Bromodichloromethan	0.456	0.474	0.462	0.510	0.527	0.486	6.43
39)	T	2-Chloroethyl Vinyl	0.192	0.188	0.198	0.241	0.235	0.211	11.94
40)	T	cis-1,3-Dichloropro	0.523	0.523	0.525	0.610	0.628	0.562	9.38
41)	T	4-Methyl-2-Pentanon	0.312	0.286	0.300	0.350	0.329	0.315	7.97
42)	S	SUR: d8-Toluene	1.370	1.275	1.176	1.260	1.208	1.258	5.89
43)	I	IS: d5-Chlorobenzene	----------	----------	ISTD	----------	----------		
44)	TCM	Toluene	2.854	2.856	2.738	3.448	3.261	3.031	10.09
45)	T	trans-1,3-Dichlorop	0.804	0.824	0.856	1.056	0.994	0.907	12.31
46)	T	1,1,2-Trichloroetha	0.365	0.362	0.369	0.468	0.401	0.393	11.38
47)	T	Tetrachloroethene	0.539	0.540	0.537	0.588	0.604	0.562	5.69
48)	T	1,3-Dichloropropane	0.603	0.577	0.595	0.769	0.658	0.640	12.12
49)	T	2-Hexanone	0.427	0.431	0.434	0.513	0.479	0.457	8.36
50)	T	Dibromochloromethan	0.620	0.615	0.627	0.783	0.742	0.677	11.69
51)	T	1,2-Dibromoethane	0.553	0.557	0.583	0.715	0.681	0.618	12.14
52)	TPM	Chlorobenzene	1.701	1.726	1.679	2.089	2.018	1.842	10.57
53)	T	1,1,1,2-Tetrachloro	0.726	0.710	0.730	0.874	0.846	0.777	9.84
54)	TC	Ethyl Benzene	2.998	3.083	3.042	3.714	3.659	3.299	10.78
55)	T	m/p-Xylene	2.839	2.833	2.864	3.352	2.980	2.974	7.38
56)	T	o-Xylene	2.697	2.783	2.858	3.424	3.110	2.974	9.91
57)	T	Styrene	1.635	1.712	1.675	2.108	2.082	1.843	12.61
58)	TP	Bromoform	0.366	0.359	0.367	0.453	0.434	0.396	11.15
59)	T	Isopropylbenzene	3.230	3.253	3.213	3.919	3.793	3.482	9.91
60)	S	Sur: 4-Bromofluorob	1.161	1.110	0.974	1.126	1.078	1.090	6.53
61)	I	IS: d4-1,4-Dichlorobe	----------	----------	ISTD	----------	----------		
62)	T	1,4-trans-2-dichlor	0.299	0.284	0.303	0.399	0.415	-----	
							QO	A= 0.015	R=0.999
								B= 0.341	
63)	T	Bromobenzene	1.245	1.200	1.245	1.547	1.386	1.325	10.78
64)	TP	1,1,2,2-Tetrachloro	0.909	0.854	0.899	1.109	1.095	0.973	12.25
65)	T	1,2,3-Trichloroprop	1.435	1.201	1.293	1.683	1.569	1.436	13.68
66)	T	n-Propylbenzene	5.133	5.018	5.073	6.214	6.204	5.528	11.27
67)	T	2-Chlorotoluene	1.178	1.170	1.191	1.448	1.437	1.285	11.22
68)	T	1,3,5-Trimethylbenz	4.270	4.071	4.114	4.990	5.019	4.493	10.53
69)	T	4-Chlorotoluene	3.812	3.701	3.801	4.725	4.621	4.132	12.03
70)	T	tert-Butylbenzene	3.927	3.977	4.032	4.733	4.857	4.305	10.47
71)	T	1,2,4-Trimethylbenz	4.114	3.994	3.884	5.020	4.971	4.397	12.58
72)	T	sec-Butylbenzene	4.751	4.802	4.828	5.901	5.884	5.233	11.51
73)	T	1,3-Dichlorbenzene	2.005	1.960	2.078	2.451	2.523	2.203	11.97
74)	T	4-Isopropyltoluene	4.222	4.263	4.175	5.001	5.150	4.562	10.38
75)	T	1,4-Dichlorobenzene	2.056	1.976	1.897	2.444	2.313	2.137	10.86
76)	T	1,2-Dichlorobenzene	1.958	1.787	1.783	1.978	1.862	1.874	4.91
77)	T	n-Butylbenzene	4.008	3.955	4.026	4.765	4.883	4.327	10.5

L = Linear LO = Linear+Origin Q = Quad QO = Quad+Origin R = Corr. Coef
(#) = Out of Range

Response Factor Report

Method : C:\SAT132\QUANT\8260B-3.M (RTE Integrator)
Title : Saturn 132
Last Update : Fri Feb 26 15:11:05 1999
Response via : Initial Calibration

Calibration Files
low =CAP0301.D mlow =CBP0301.D mid =CCP0301.D
mhi =CDP0301.D high =CEP0301.D =

		Compound	low	mlow	mid	mhi	high	Avg	%RSD
78)	T	1,2-Dibromo-3-chlor	0.349	0.311	0.344	0.436	0.413	0.371	14.00
79)	T	1,2,4-Trichlorobenz	1.291	1.199	1.125	1.383	1.336	1.267	8.23
80)	T	Hexachlorobutadiene	1.118	1.025	1.039	1.248	1.192	1.124	8.58
81)	T	Naphthalene	3.060	2.518	2.643	3.244	2.922	2.878	10.33
82)	T	1,2,3-Trichlorobenz	1.250	1.123	1.114	1.312	1.332	1.226	8.42

L = Linear LO = Linear+Origin Q = Quad QO = Quad+Origin R = Corr. Coef
(#) = Out of Range

Compound List Report

Method : C:\SAT132\QUANT\8260B-3.M (RTE Integrator)
Title : Saturn 132
Last Update : Fri Feb 26 15:11:05 1999
Response via : Initial Calibration
Total Cpnds : 82

PK#			Compound Name	QIon	Exp_RT	Rel_RT	Cal	#Qual	A/H	ID
1	I		IS: Flourobenzene	96	16.15	1.000	A	2	A	B
2		T	Dichlorodifluoromethane	85	2.97	0.184	A	2	A	B
3		T	Chloromethane	50	3.88	0.240	A	2	A	B
4		T	Vinyl Chloride	62	4.45	0.276	A	2	A	B
5		T	Bromomethane	94	5.88	0.364	QO	2	A	B
6		T	Chloroethane	45	6.40	0.396	A	2	A	B
7		T	Trichlorofluoromethane	101	7.43	0.460	A	2	A	B
8		T	Ethyl Ether	45	8.63	0.534	A	2	A	B
9		T	Acrolein	55	8.92	0.552	A	2	A	B
10		T	1,1-Dichloroethene	96	9.08	0.562	A	2	A	B
11		T	Trichlorotrifluoroethane	101	9.28	0.575	A	2	A	B
12		T	Iodomethane	142	9.36	0.580	LO	2	A	B
13		T	Carbon Disulfide	76	9.43	0.584	A	2	A	B
14		T	Acetone	43	9.44	0.585	LO	2	A	B
15		T	Isopropanol	45	10.29	0.638	QO	2	A	B
16		T	Methylene Chloride	49	10.56	0.654	A	2	A	B
17		T	Acrylonitrile	52	11.25	0.697	A	2	A	B
18		T	trans-1,2-Dichloroethene	96	11.25	0.697	A	2	A	B
19		T	Methyl(tert)Butyl Ether	73	11.35	0.703	A	2	A	B
20		T	1,1-Dichloroethane	63	12.27	0.760	A	2	A	B
21		T	Vinyl Acetate	43	12.55	0.777	A	2	A	B
22		T	2,2-Dichloropropane	77	13.57	0.841	A	2	A	B
23		T	cis-1,2-Dichloroethene	96	13.63	0.844	A	2	A	B
24		T	Methyl Ethyl Ketone	43	13.76	0.852	A	2	A	B
25		T	Bromochloromethane	49	14.13	0.875	A	2	A	B
26		T	Chloroform	83	14.39	0.891	A	2	A	B
27		T	1,1,1-Trichloroethane	97	14.71	0.911	A	2	A	B
28		S	SUR: Dibromofluoromethane	113	14.73	0.913	A	2	A	B
29		T	Carbon Tetrachloride	117	15.05	0.932	A	2	A	B
30		T	1,1-Dichloropropene	75	15.08	0.934	A	2	A	B
31		T	Benzene	78	15.52	0.961	A	2	A	B
32		T	1,2-Dichloroethane	62	15.59	0.965	A	2	A	B
33		T	Heptane	41	16.19	1.003	A	2	A	B
34		S	SUR: 1,2-Dichloroethane-d4	65	15.41	0.955	A	2	A	B
35		T	Trichloroethene	130	16.96	1.051	A	2	A	B
36		T	1,2-Dichloropropane	63	17.44	1.080	A	2	A	B
37		T	Dibromomethane	93	17.69	1.096	A	2	A	B
38		T	Bromodichloromethane	83	18.06	1.119	A	2	A	B
39		T	2-Chloroethyl Vinyl Ether	63	18.77	1.163	A	2	A	B
40		T	cis-1,3-Dichloropropene	75	19.07	1.181	A	2	A	B
41		T	4-Methyl-2-Pentanone	43	19.44	1.204	A	2	A	B
42		S	SUR: d8-Toluene	98	19.65	1.217	A	2	A	B
43	I		IS: d5-Chlorobenzene	82	23.03	1.000	A	2	A	B
44		T	Toluene	91	19.80	0.860	A	2	A	B
45		T	trans-1,3-Dichloropropene	75	20.33	0.883	A	2	A	B
46		T	1,1,2-Trichloroethane	83	20.75	0.901	A	2	A	B
47		T	Tetrachloroethene	129	21.07	0.915	A	2	A	B
48		T	1,3-Dichloropropane	76	21.12	0.917	A	2	A	B
49		T	2-Hexanone	43	21.36	0.928	A	2	A	B
50		T	Dibromochloromethane	129	21.65	0.940	A	2	A	B
51		T	1,2-Dibromoethane	109	21.91	0.951	A	2	A	B
52		T	Chlorobenzene	112	23.09	1.003	A	2	A	B

53	T	1,1,1,2-Tetrachloroethane	131	23.31	1.012	A	2	A	B
54	T	Ethyl Benzene	91	23.37	1.015	A	2	A	B
55	T	m/p-Xylene	91	23.68	1.028	A	2	A	B
56	T	o-Xylene	91	24.64	1.070	A	2	A	B
57	T	Styrene	104	24.68	1.072	A	2	A	B
58	T	Bromoform	173	25.13	1.092	A	2	A	B
59	T	Isopropylbenzene	105	25.59	1.111	A	2	A	B
60	S	Sur: 4-Bromofluorobenzene	95	25.97	1.128	A	2	A	B
61	I	IS: d4-1,4-Dichlorobenzene	152	28.85	1.000	A	2	A	B
62	T	1,4-trans-2-dichlorobutene	53	26.49	0.918	QO	1	A	B
63	T	Bromobenzene	156	26.33	0.913	A	2	A	B
64	T	1,1,2,2-Tetrachloroethane	83	26.33	0.913	A	2	A	B
65	T	1,2,3-Trichloropropane	75	26.44	0.916	A	2	A	B
66	T	n-Propylbenzene	91	26.63	0.923	A	2	A	B
67	T	2-Chlorotoluene	126	26.84	0.930	A	2	A	B
68	T	1,3,5-Trimethylbenzene	105	27.09	0.939	A	2	A	B
69	T	4-Chlorotoluene	91	27.12	0.940	A	2	A	B
70	T	tert-Butylbenzene	119	27.92	0.968	A	2	A	B
71	T	1,2,4-Trimethylbenzene	105	28.05	0.972	A	2	A	B
72	T	sec-Butylbenzene	105	28.45	0.986	A	2	A	B
73	T	1,3-Dichlorbenzene	146	28.71	0.995	A	2	A	B
74	T	4-Isopropyltoluene	119	28.80	0.998	A	2	A	B
75	T	1,4-Dichlorobenzene	146	28.89	1.001	A	2	A	B
76	T	1,2-Dichlorobenzene	146	29.72	1.030	A	2	A	B
77	T	n-Butylbenzene	91	29.71	1.030	A	2	A	B
78	T	1,2-Dibromo-3-chloropropane	75	31.30	1.085	A	2	A	B
79	T	1,2,4-Trichlorobenzene	180	33.08	1.147	A	2	A	B
80	T	Hexachlorobutadiene	225	33.47	1.160	A	2	A	B
81	T	Naphthalene	128	33.64	1.166	A	2	A	B
82	T	1,2,3-Trichlorobenzene	180	34.20	1.185	A	2	A	B

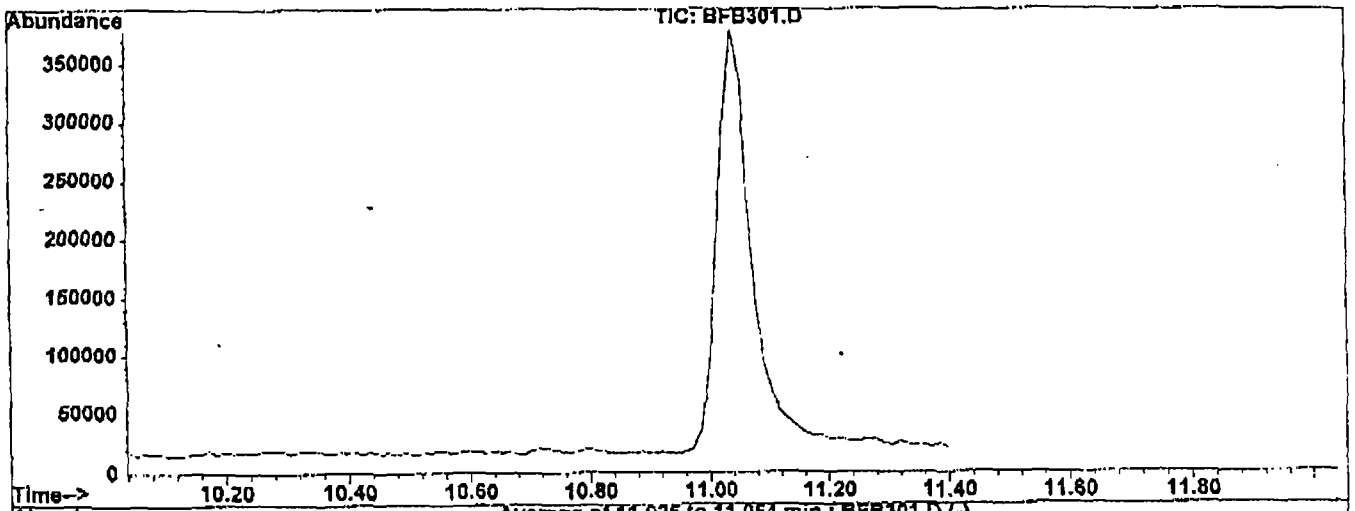
Cal A = Average L = Linear LO = Linear w/origin Q = Quad QO = Quad w/orig
#Qual = number of qualifiers
A/H = Area or Height
ID R = R.T. B = R.T. & Q Q = Qvalue L = Largest A = All

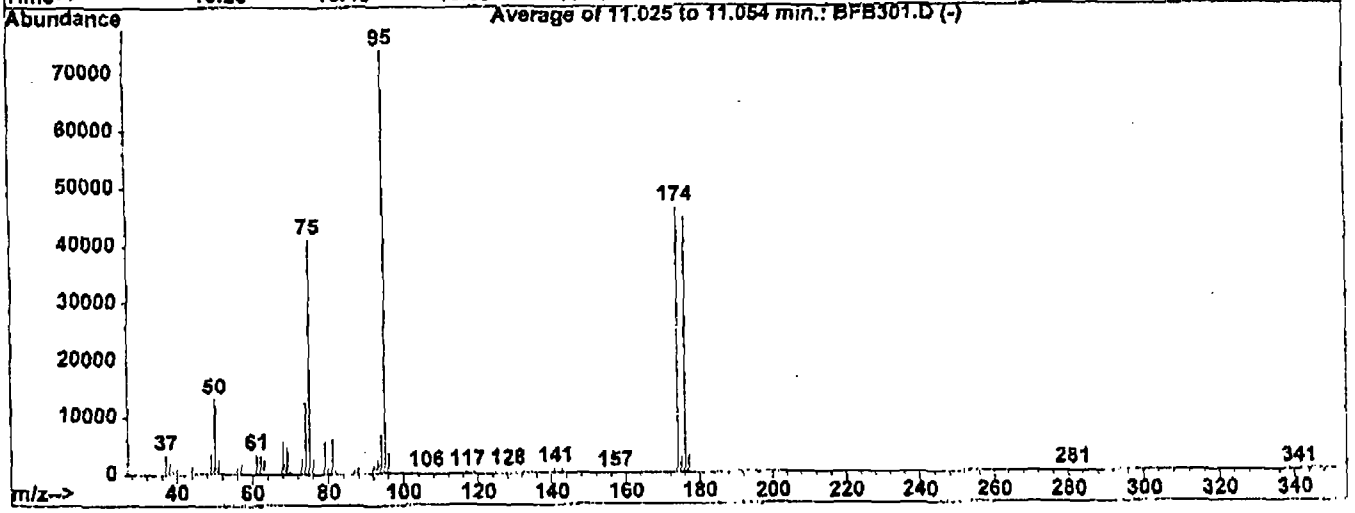
8260B-3.M Tue Mar 02 09:46:04 1999

CLPBFB Tune Evaluation

Data File : C:\HPCHEM\1\DATA\BFB301.D
Acq On : 1 MAR 1999 11:00
Sample :
Misc :
MS Integration Params: rteint.p
Method : C:\SAT132\QUANT\8260B-3.M (RTE Integrator)
Title : Saturn 132

Vial: 0
Operator:
Inst :
Multiplr: 1.00





Peak Apex is scan: 828 (11.04 min)
Average of 3 scans: 827,828,829 minus background scan 808 (10.77 min)

Target Mass	Rel. to Mass	Lower Limit,%	Upper Limit,%	Rel. Abn,%	Raw Abn	Result Pass/Fail
50	95	15	40	18.0	13330	PASS
75	95	30	60	55.5	41097	PASS
95	95	100	100	100.0	74088	PASS
96	95	5	9	5.1	3805	PASS
173	174	0	2	1.0	451	PASS
174	95	50	100	62.5	46309	PASS
175	174	5	9	6.5	3016	PASS
176	174	95	101	96.4	44653	PASS
177	176	5	9	6.7	3014	PASS

BFB301.D 8260B-3.M Mon Mar 01 11:15:18 1999

Average of 11.025 to 11.054 min.: BFB301.D

Modified:subtracted

m/z	abund.	m/z	abund.	m/z	abund.	m/z	abund.
37.00	3432	57.00	2092	76.00	2915	95.00	74088
38.00	2107	60.00	772	79.00	5716	96.00	3805
39.00	658	61.00	3507	80.00	1094	104.00	425
40.00	1093	62.00	3315	81.00	6102	117.00	376
44.00	1428	63.00	2562	82.00	841	119.00	378
45.00	506	68.00	6107	87.00	782	128.00	492
48.00	511	69.00	4755	88.00	1080	141.00	942
49.00	3648	70.00	596	91.00	420	143.00	844
50.00	13330	73.00	3058	92.00	1763	173.00	451
51.00	2731	74.00	12716	93.00	2657	174.00	46309
56.00	1399	75.00	41097	94.00	7112	175.00	3016

Average of 11.025 to 11.054 min.: BFB301.D

Modified:subtracted

m/z	abund.	m/z	abund.	m/z	abund.	m/z	abund.
176.00	44653						
177.00	3014						

Quantitation Report

Data File : C:\HPCHEM\1\DATA\CAP0301.D Vial: 0
Acq On : 1 MAR 1999 12:12 Operator:
Sample : Inst :
Misc : Multiplr: 1.00
MS Integration Params: rteint.p
Quant Time: Mar 2 9:20 1999 Quant Results File: 8260B-3.RES

Quant Method : c:\sat132\quant\8260b-3.m (RTE Integrator)
Title : Saturn 132
Last Update : Fri Feb 26 15:11:05 1999
Response via : Initial Calibration
DataAcq Meth :
IS QA File : C:\HPCHEM\1\DATA\CCP0226.D (26 FEB 1999 12:14)

Internal Standards	R.T.	QIon	Response	Conc	Units		Dev(Min) Rcv(Ar)
1) IS: Flourobenzene	16.13	96	1291740	40.00	ug/L		-0.01 97.45%
43) IS: d5-Chlorobenzene	23.04	82	749960	40.00	ug/L		0.01 98.77%
61) IS: d4-1,4-Dichlorobenzene	28.85	152	548691	40.00	ug/L		0.00 92.46%
System Monitoring Compounds							
28) SUR: Dibromofluoromethane	14.71	113	955431	90.81	ug/L		-0.03
Spiked Amount 40.000			Recovery = 227.03%				
34) SUR: 1,2-Dichloroethane-d4	15.39	65	507532	41.22	ug/L		-0.03
Spiked Amount 40.000			Recovery = 103.05%				
42) SUR: d8-Toluene	19.64	98	3538515	89.07	ug/L		-0.01
Spiked Amount 40.000			Recovery = 222.68%				
60) Sur: 4-Bromofluorobenzene	25.97	95	1741462	85.35	ug/L		0.00
Spiked Amount 40.000			Recovery = 213.38%				
Target Compounds							Qvalue
2) Dichlorodifluoromethane	2.95	85	48017	8.25	ug/L		100
3) Chloromethane	3.89	50	38827	8.23	ug/L		96
4) Vinyl Chloride	4.45	62	62812	9.67	ug/L		100
5) Bromomethane	5.88	94	69342	9.90	ug/L		99
6) Chloroethane	6.38	45	6666	9.72	ug/L	#	31
7) Trichlorofluoromethane	7.39	101	152056	9.64	ug/L		97
8) Ethyl Ether	8.64	45	41189	9.14	ug/L		99
9) Acrolein	8.91	55	6240m	12.38	ug/L		99
10) 1,1-Dichloroethene	9.05	96	80835	9.42	ug/L		100
11) Trichlorotrifluoroethane	9.25	101	118582	9.60	ug/L	#	78
12) Iodomethane	9.35	142	167581m	8.28	ug/L		97
13) Carbon Disulfide	9.40	76	183145	10.02	ug/L	#	100
14) Acetone	9.45	43	56999	16.80	ug/L		97
15) Isopropanol	10.32	45	4021	14.99	ug/L	#	87
16) Methylene Chloride	10.53	49	195760	10.01	ug/L	#	98
17) Acrylonitrile	11.23	52	50411	12.27	ug/L	#	91
18) trans-1,2-Dichloroethene	11.21	96	91261	9.95	ug/L		99
19) Methyl(tert)Butyl Ether	11.32	73	266552	9.53	ug/L		96
20) 1,1-Dichloroethane	12.24	63	128906	9.37	ug/L		99
21) Vinyl Acetate	12.55	43	141148	9.71	ug/L		99
22) 2,2-Dichloropropane	13.55	77	168263	9.43	ug/L		99
23) cis-1,2-Dichloroethene	13.60	96	102702	10.01	ug/L		98
24) Methyl Ethyl Ketone	13.75	43	55356	13.23	ug/L	#	96
25) Bromochloromethane	14.11	49	122853	9.76	ug/L		99
26) Chloroform	14.36	83	187173	9.55	ug/L		98
27) 1,1,1-Trichloroethane	14.67	97	208386	9.50	ug/L		99

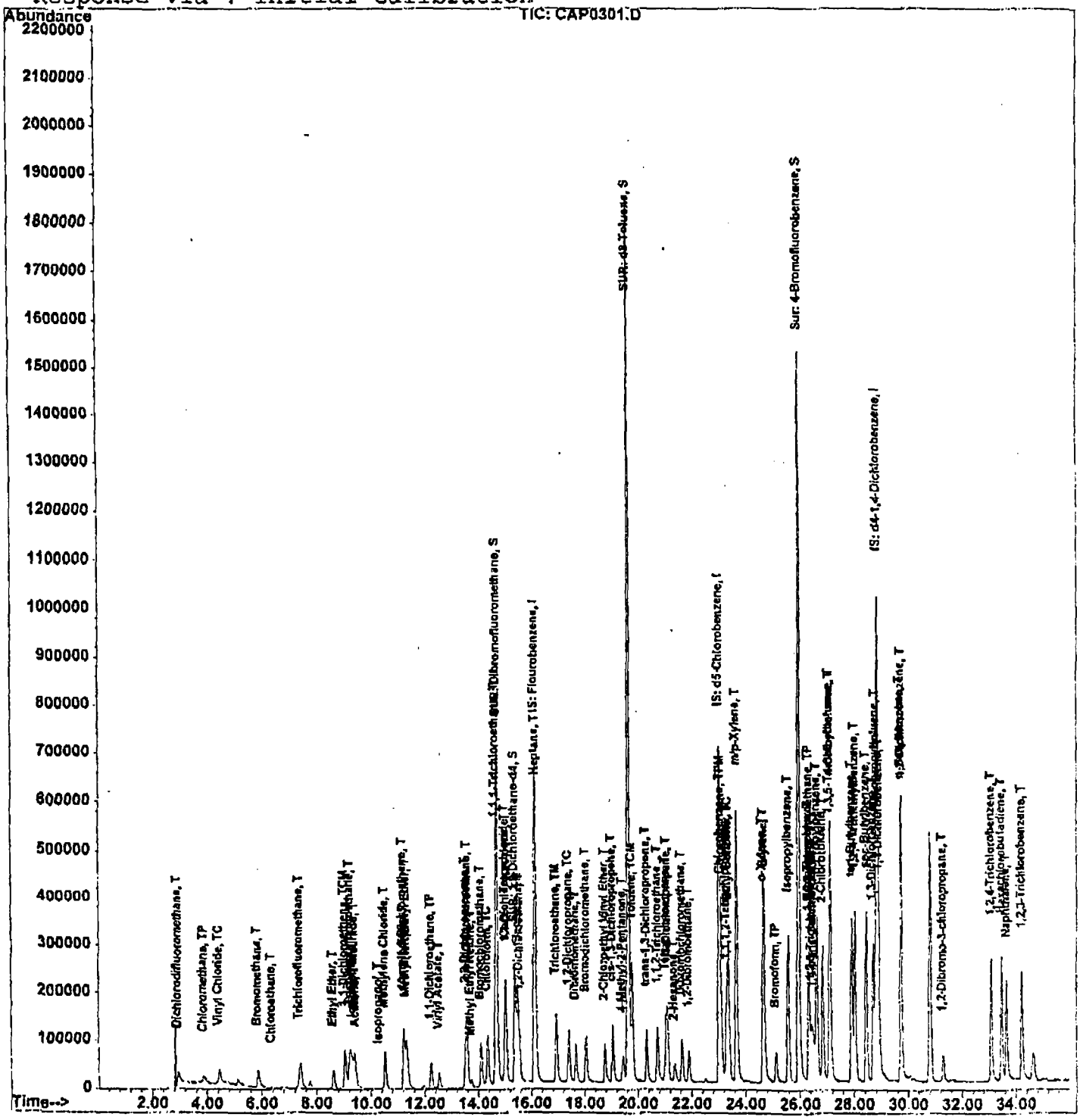
29)	Carbon Tetrachloride	15.04	117	155352	9.55 ug/L	99
30)	1,1-Dichloropropene	15.05	75	147727	9.57 ug/L	97
31)	Benzene	15.49	78	399271	9.69 ug/L	97
32)	1,2-Dichloroethane	15.56	62	161540	10.07 ug/L	99
33)	Heptane	16.16	41	182685	9.99 ug/L #	82
35)	Trichloroethene	16.93	130	105993	9.84 ug/L	98
36)	1,2-Dichloropropane	17.41	63	74281	10.22 ug/L	9[illegible]
37)	Dibromomethane	17.68	93	82517	9.84 ug/L	98
38)	Bromodichloromethane	18.07	83	147119	9.62 ug/L	99
39)	2-Chloroethyl Vinyl Ether	18.77	63	62148	9.44 ug/L	98
40)	cis-1,3-Dichloropropene	19.05	75	168738	9.75 ug/L	99
41)	4-Methyl-2-Pentanone	19.44	43	100727	10.35 ug/L	99
44)	Toluene	19.80	91	535150	9.77 ug/L	99
45)	trans-1,3-Dichloropropene	20.33	75	150768	9.27 ug/L	98
46)	1,1,2-Trichloroethane	20.75	83	68491	9.61 ug/L	97
47)	Tetrachloroethene	21.07	129	101101	9.78 ug/L	97
48)	1,3-Dichloropropane	21.11	76	113138	9.98 ug/L	89
49)	2-Hexanone	21.39	43	79993	11.08 ug/L	98
50)	Dibromochloromethane	21.65	129	116293	9.62 ug/L	99
51)	1,2-Dibromoethane	21.91	109	103709	9.29 ug/L	98
52)	Chlorobenzene	23.09	112	318841	9.57 ug/L	95
53)	1,1,1,2-Tetrachloroethane	23.31	131	136077	9.73 ug/L	98
54)	Ethyl Benzene	23.37	91	562128	9.34 ug/L	98
55)	m/p-Xylene	23.67	91	1064695	19.70 ug/L	98
56)	o-Xylene	24.64	91	505660	9.16 ug/L	97
57)	Styrene	24.69	104	306520	9.14 ug/L	92
58)	Bromoform	25.13	173	68642	9.71 ug/L	98
59)	Isopropylbenzene	25.59	105	605632	9.93 ug/L	99
62)	1,4-trans-2-dichlorobutene	26.49	53	40947	9.31 ug/L	98
63)	Bromobenzene	26.33	156	170736	9.82 ug/L	98
64)	1,1,2,2-Tetrachloroethane	26.35	83	124687	10.03 ug/L #	88
65)	1,2,3-Trichloropropane	26.44	75	196840	9.99 ug/L	97
66)	n-Propylbenzene	26.63	91	704041	9.64 ug/L	9[illegible]
67)	2-Chlorotoluene	26.84	126	161524	9.87 ug/L	9[illegible]
68)	1,3,5-Trimethylbenzene	27.09	105	585743	9.99 ug/L	96
69)	4-Chlorotoluene	27.11	91	522891	9.65 ug/L	99
70)	tert-Butylbenzene	27.93	119	538645	9.49 ug/L #	97
71)	1,2,4-Trimethylbenzene	28.05	105	564381	9.49 ug/L	100
72)	sec-Butylbenzene	28.45	105	651737	9.55 ug/L #	96
73)	1,3-Dichlorbenzene	28.71	146	274969	9.62 ug/L	100
74)	4-Isopropyltoluene	28.80	119	579080	9.86 ug/L	97
75)	1,4-Dichlorobenzene	28.92	146	282039	10.05 ug/L	94
76)	1,2-Dichlorobenzene	29.73	146	268616	10.85 ug/L	98
77)	n-Butylbenzene	29.71	91	549780	9.92 ug/L #	94
78)	1,2-Dibromo-3-chloropropan	31.30	75	47871	10.01 ug/L	97
79)	1,2,4-Trichlorobenzene	33.09	180	177112	11.06 ug/L	96
80)	Hexachlorobutadiene	33.48	225	153333	10.45 ug/L	99
81)	Naphthalene	33.65	128	419735	11.84 ug/L	99
82)	1,2,3-Trichlorobenzene	34.23	180	171415	10.86 ug/L	100

(#) = qualifier out of range (m) = manual integration
CAP0301.D 8260B-3.M Tue Mar 02 09:53:54 1999

Quantitation Report

Data File : c:\hpchem\1\data\CAP0301.D Vial: 0
Acq On : 1 MAR 1999 12:12 Operator:
Sample : Inst :
Misc : Multiplr: 1.00
MS Integration Params: rteint.p
Quant Time: Mar 1 12:50 1999 Quant Results File: 8260b-3.RES

Method : c:\sat132\quant\8260b-3.m (RTE Integrator)
Title : Saturn 132
Last Update : Fri Feb 26 15:11:05 1999
Response via : Initial Calibration



Quantitation Report

Data File : C:\HPCHEM\1\DATA\CBP0301.D Vial: 0
Acq On : 1 MAR 1999 12:54 Operator:
Sample : Inst :
Misc : Multiplr: 1.00
MS Integration Params: rteint.p
Quant Time: Mar 1 13:40 1999 Quant Results File: 8260B-3.RES

Quant Method : c:\sat132\quant\8260b-3.m (RTE Integrator)
Title : Saturn 132
Last Update : Fri Feb 26 15:11:05 1999
Response via : Initial Calibration
DataAcq Meth :
IS QA File : C:\HPCHEM\1\DATA\CCP0226.D (26 FEB 1999 12:14)

Internal Standards	R.T.	QIon	Response	Conc	Units	Dev(Min) Rcv(Ar)
1) IS: Flourobenzene	16.13	96	1303235	40.00	ug/L	-0.01 98.32%
43) IS: d5-Chlorobenzene	23.03	82	744523	40.00	ug/L	0.00 98.06%
61) IS: d4-1,4-Dichlorobenzene	28.85	152	546033	40.00	ug/L	0.00 92.01%
System Monitoring Compounds						
28) SUR: Dibromofluoromethane	14.71	113	788895	74.32	ug/L	-0.03
Spiked Amount 40.000			Recovery =	185.80%		
34) SUR: 1,2-Dichloroethane-d4	15.40	65	504697	40.63	ug/L	-0.01
Spiked Amount 40.000			Recovery =	101.58%		
42) SUR: d8-Toluene	19.65	98	2906788	72.52	ug/L	0.00
Spiked Amount 40.000			Recovery =	181.30%		
60) Sur: 4-Bromofluorobenzene	25.96	95	1446290	71.40	ug/L	-0.0
Spiked Amount 40.000			Recovery =	178.50%		
Target Compounds						Qvalue
2) Dichlorodifluoromethane	2.96	85	112278	19.12	ug/L	97
3) Chloromethane	3.89	50	85371	17.94	ug/L	99
4) Vinyl Chloride	4.45	62	127179	19.40	ug/L	96
5) Bromomethane	5.88	94	125874	17.80	ug/L	100
6) Chloroethane	6.40	45	13805	19.94	ug/L #	42
7) Trichlorofluoromethane	7.40	101	309930	19.49	ug/L	96
8) Ethyl Ether	8.63	45	91500	20.12	ug/L	96
9) Acrolein	8.93	55	10784	21.21	ug/L	94
10) 1,1-Dichloroethene	9.06	96	168862	19.50	ug/L	97
11) Trichlorotrifluoroethane	9.25	101	244269	19.60	ug/L #	80
12) Iodomethane	9.35	142	398937	19.53	ug/L	98
13) Carbon Disulfide	9.41	76	365257	19.82	ug/L #	99
14) Acetone	9.44	43	95252	27.83	ug/L	98
15) Isopropanol	10.28	45	6508	24.06	ug/L #	60
16) Methylene Chloride	10.53	49	389283	19.73	ug/L	98
17) Acrylonitrile	11.24	52	79786	19.25	ug/L	96
18) trans-1,2-Dichloroethene	11.23	96	187561	20.26	ug/L	98
19) Methyl(tert)Butyl Ether	11.33	73	531422	18.83	ug/L	97
20) 1,1-Dichloroethane	12.24	63	270077	19.47	ug/L	99
21) Vinyl Acetate	12.53	43	290097	19.77	ug/L	100
22) 2,2-Dichloropropane	13.55	77	301430m	16.74	ug/L	26
23) cis-1,2-Dichloroethene	13.59	96	208214	20.12	ug/L	98
24) Methyl Ethyl Ketone	13.74	43	106385	25.20	ug/L	
25) Bromochloromethane	14.11	49	252790	19.90	ug/L	
26) Chloroform	14.36	83	394039	19.92	ug/L	99
27) 1,1,1-Trichloroethane	14.67	97	423870	19.15	ug/L	100

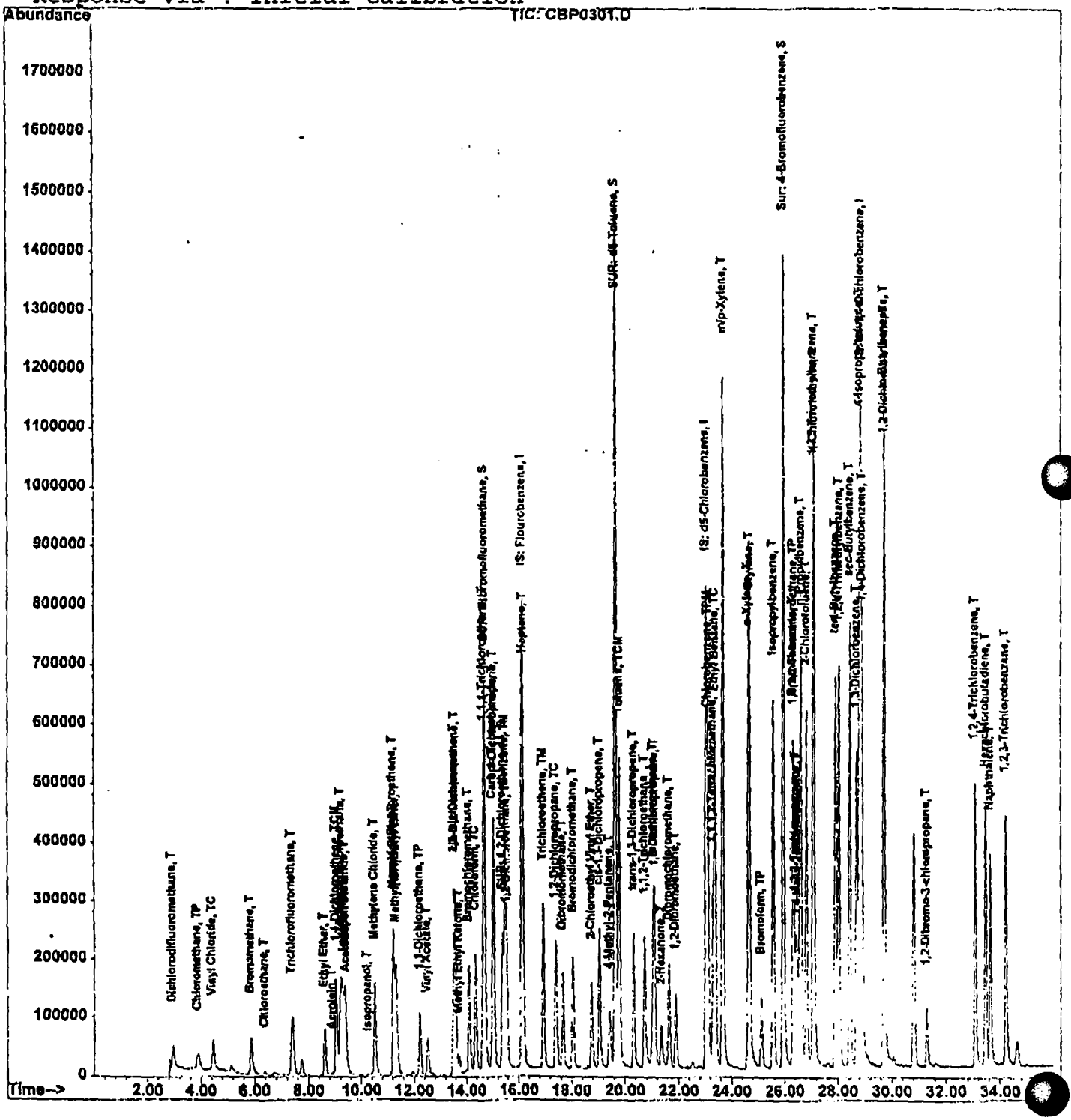
29) Carbon Tetrachloride	15.03	117	316803	19.31	ug/L	
30) 1,1-Dichloropropene	15.05	75	303591	19.50	ug/L	100
31) Benzene	15.49	78	797673	19.19	ug/L	99
32) 1,2-Dichloroethane	15.56	62	316428	19.55	ug/L	99
33) Heptane	16.17	41	374193	20.29	ug/L	95
35) Trichloroethene	16.95	130	214015	19.69	ug/L	99
36) 1,2-Dichloropropane	17.41	63	145938	19.91	ug/L	99
37) Dibromomethane	17.68	93	162567	19.21	ug/L	97
38) Bromodichloromethane	18.06	83	308753	20.02	ug/L	99
39) 2-Chloroethyl Vinyl Ether	18.77	63	122293	18.41	ug/L #	98
40) cis-1,3-Dichloropropene	19.07	75	340510	19.49	ug/L	99
41) 4-Methyl-2-Pentanone	19.44	43	186148	18.96	ug/L	98
44) Toluene	19.80	91	1063028	19.55	ug/L	100
45) trans-1,3-Dichloropropene	20.33	75	306730	19.01	ug/L	99
46) 1,1,2-Trichloroethane	20.75	83	134787	19.04	ug/L	94
47) Tetrachloroethene	21.06	129	200945	19.58	ug/L	98
48) 1,3-Dichloropropane	21.12	76	214924	19.09	ug/L	91
49) 2-Hexanone	21.39	43	160437	22.38	ug/L	97
50) Dibromochloromethane	21.65	129	228904	19.07	ug/L	98
51) 1,2-Dibromoethane	21.91	109	207436	18.71	ug/L	98
52) Chlorobenzene	23.11	112	642381	19.43	ug/L	96
53) 1,1,1,2-Tetrachloroethane	23.31	131	264332	19.05	ug/L	99
54) Ethyl Benzene	23.39	91	1147575	19.21	ug/L	97
55) m/p-Xylene	23.68	91	2109272	39.31	ug/L	99
56) o-Xylene	24.64	91	1035976	18.89	ug/L	99
57) Styrene	24.69	104	637492	19.16	ug/L	97
58) Bromoform	25.13	173	133473	19.03	ug/L	98
59) Isopropylbenzene	25.59	105	1211088	20.01	ug/L	99
62) 1,4-trans-2-dichlorobutene	26.51	53	77537	17.71	ug/L	96
63) Bromobenzene	26.33	156	327626	18.94	ug/L	97
64) 1,1,2,2-Tetrachloroethane	26.35	83	233140	18.85	ug/L	100
65) 1,2,3-Trichloropropane	26.44	75	327878	16.73	ug/L #	93
66) n-Propylbenzene	26.63	91	1369916	18.85	ug/L	98
67) 2-Chlorotoluene	26.84	126	319438	19.62	ug/L	95
68) 1,3,5-Trimethylbenzene	27.09	105	1111325	19.05	ug/L	97
69) 4-Chlorotoluene	27.12	91	1010352	18.73	ug/L	98
70) tert-Butylbenzene	27.92	119	1085919	19.22	ug/L #	99
71) 1,2,4-Trimethylbenzene	28.05	105	1090418	18.43	ug/L	98
72) sec-Butylbenzene	28.45	105	1311000	19.31	ug/L #	96
73) 1,3-Dichlorbenzene	28.71	146	535152	18.81	ug/L	99
74) 4-Isopropyltoluene	28.81	119	1163870	19.90	ug/L	99
75) 1,4-Dichlorobenzene	28.91	146	539405	19.31	ug/L	99
76) 1,2-Dichlorobenzene	29.73	146	487838	19.80	ug/L	99
77) n-Butylbenzene	29.71	91	1079745	19.57	ug/L	97
78) 1,2-Dibromo-3-chloropropan	31.32	75	84928	17.84	ug/L	99
79) 1,2,4-Trichlorobenzene	33.08	180	327298	20.53	ug/L	99
80) Hexachlorobutadiene	33.49	225	279733	19.15	ug/L #	87
81) Naphthalene	33.65	128	687577	19.49	ug/L	99
82) 1,2,3-Trichlorobenzene	34.23	180	306518	19.51	ug/L	99

(#) = qualifier out of range (m) = manual integration
CBP0301.D 8260B-3.M Tue Mar 02 09:55:09 1999

Quantitation Report

Data File : c:\hpchem\1\data\CBP0301.D Vial: 0
Acq On : 1 MAR 1999 12:54 Operator:
Sample : Inst :
Misc : Multiplr: 1.00
MS Integration Params: rteint.p
Quant Time: Mar 1 13:32 1999 Quant Results File: 8260b-3.RES

Method : c:\sat132\quant\8260b-3.m (RTE Integrator)
Title : Saturn 132
Last Update : Fri Feb 26 15:11:05 1999
Response via : Initial Calibration



Quantitation Report

Data File : C:\HPCHEM\1\DATA\CCP0301.D Vial: 0
Acq On : 1 MAR 1999 13:36 Operator:
Sample : Inst :
Misc : Multiplr: 1.00
MS Integration Params: rteint.p
Quant Time: Mar 1 14:13 1999 Quant Results File: 8260B-3.RES

Quant Method : c:\sat132\quant\8260b-3.m (RTE Integrator)
Title : Saturn 132
Last Update : Fri Feb 26 15:11:05 1999
Response via : Initial Calibration
DataAcq Meth :
IS QA File : C:\HPCHEM\1\DATA\CCP0226.D (26 FEB 1999 12:14)

Internal Standards	R.T.	QIon	Response	Conc	Units		Dev(Min) Rcv(Ar)
1) IS: Flourobenzene	16.13	96	1287663	40.00	ug/L		-0.01 97.14%
43) IS: d5-Chlorobenzene	23.04	82	732174	40.00	ug/L		0.01 96.43%
61) IS: d4-1,4-Dichlorobenzene	28.85	152	530711	40.00	ug/L		0.00 89.43%
System Monitoring Compounds							
28) SUR: Dibromofluoromethane	14.69	113	390995	37.28	ug/L		-0.04
Spiked Amount 40.000			Recovery =	93.20%			
34) SUR: 1,2-Dichloroethane-d4	15.39	65	492972	40.16	ug/L		-0.03
Spiked Amount 40.000			Recovery =	100.40%			
42) SUR: d8-Toluene	19.65	98	1514484	38.24	ug/L		0.00
Spiked Amount 40.000			Recovery =	95.60%			
60) Sur: 4-Bromofluorobenzene	25.96	95	713299	35.81	ug/L		-0.01
Spiked Amount 40.000			Recovery =	89.53%			
Target Compounds							Qvalue
2) Dichlorodifluoromethane	2.97	85	227159	39.16	ug/L		96
3) Chloromethane	3.89	50	173138	36.82	ug/L		99
4) Vinyl Chloride	4.45	62	259529	40.06	ug/L		98
5) Bromomethane	5.88	94	267222	38.26	ug/L		99
6) Chloroethane	6.39	45	28132	41.13	ug/L	#	48
7) Trichlorofluoromethane	7.40	101	625501	39.80	ug/L		97
8) Ethyl Ether	8.61	45	182531	40.62	ug/L		95
9) Acrolein	8.92	55	20373	40.55	ug/L		96
10) 1,1-Dichloroethene	9.05	96	339907	39.74	ug/L		98
11) Trichlorotrifluoroethane	9.24	101	493936	40.12	ug/L	#	78
12) Iodomethane	9.35	142	850987	42.16	ug/L		99
13) Carbon Disulfide	9.41	76	732416	40.22	ug/L		98
14) Acetone	9.41	43	156100	46.15	ug/L	#	98
15) Isopropanol	10.27	45	11291	42.24	ug/L	#	90
16) Methylene Chloride	10.53	49	772487	39.62	ug/L		100
17) Acrylonitrile	11.23	52	164905	40.27	ug/L		98
18) trans-1,2-Dichloroethene	11.23	96	381073	41.66	ug/L		97
19) Methyl(tert)Butyl Ether	11.32	73	1107185	39.71	ug/L		99
20) 1,1-Dichloroethane	12.23	63	557055	40.64	ug/L		99
21) Vinyl Acetate	12.52	43	602819	41.58	ug/L		100
22) 2,2-Dichloropropane	13.55	77	706451	39.71	ug/L		97
23) cis-1,2-Dichloroethene	13.60	96	413855	40.47	ug/L		97
24) Methyl Ethyl Ketone	13.73	43	192177	46.08	ug/L	#	97
25) Bromochloromethane	14.11	49	500951	39.91	ug/L		99
26) Chloroform	14.36	83	779630	39.89	ug/L		99
27) 1,1,1-Trichloroethane	14.67	97	867861	39.69	ug/L		99

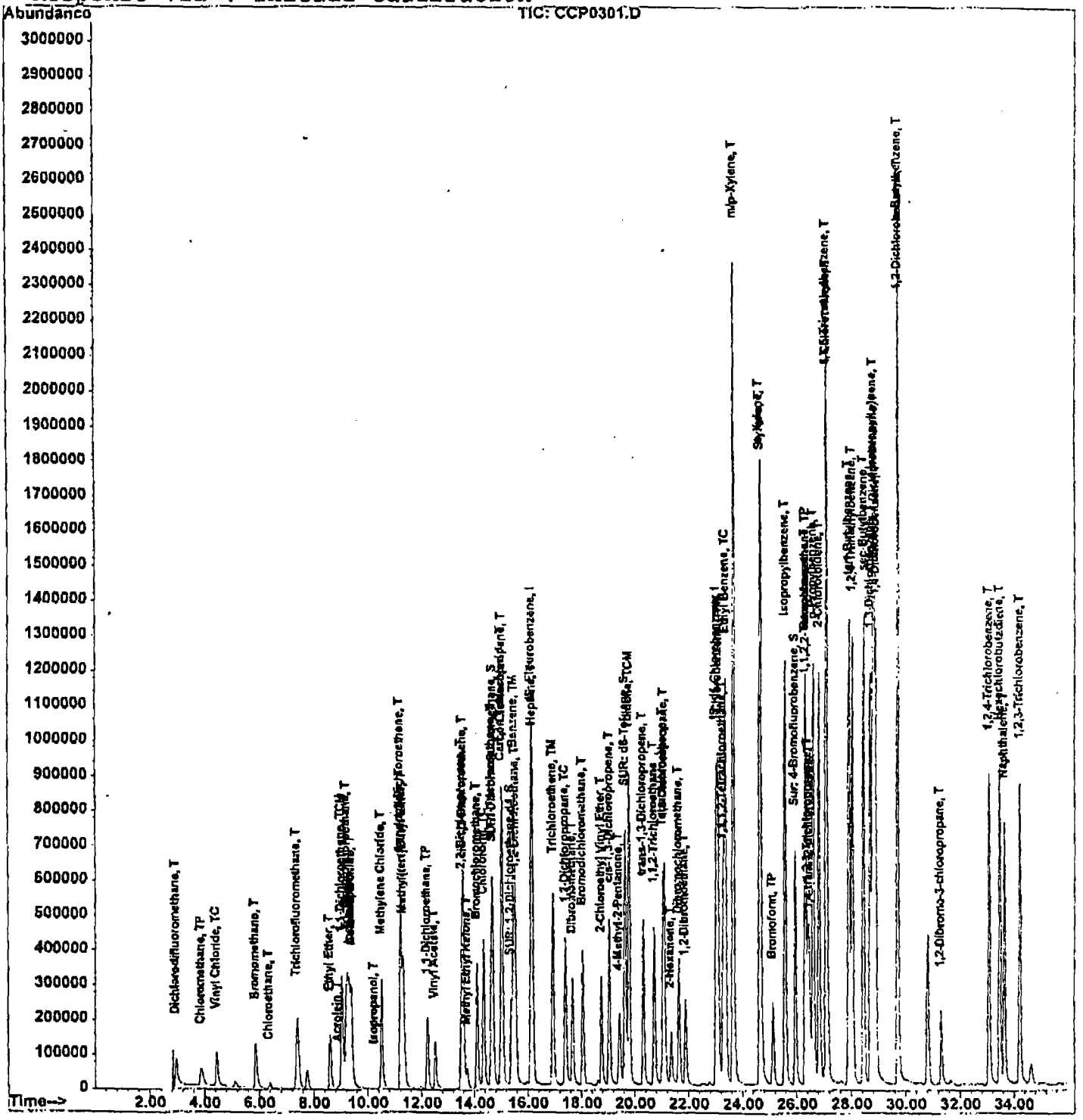
29)	Carbon Tetrachloride	15.03	117	645899	39.85	ug/L	99
30)	1,1-Dichloropropene	15.04	75	593895	38.61	ug/L	98
31)	Benzene	15.49	78	1597216	38.89	ug/L	98
32)	1,2-Dichloroethane	15.55	62	639753	40.01	ug/L	98
33)	Heptane	16.17	41	724975	39.78	ug/L	96
35)	Trichloroethene	16.95	130	429107	39.96	ug/L	97
36)	1,2-Dichloropropane	17.41	63	287346	39.68	ug/L	99
37)	Dibromomethane	17.68	93	325039	38.88	ug/L	98
38)	Bromodichloromethane	18.05	83	595272	39.06	ug/L	99
39)	2-Chloroethyl Vinyl Ether	18.76	63	255228	38.90	ug/L #	97
40)	cis-1,3-Dichloropropene	19.07	75	676195	39.18	ug/L	99
41)	4-Methyl-2-Pentanone	19.43	43	385705	39.75	ug/L	99
44)	Toluene	19.79	91	2004465	37.49	ug/L	99
45)	trans-1,3-Dichloropropene	20.32	75	626826	39.50	ug/L	99
46)	1,1,2-Trichloroethane	20.73	83	270476	38.86	ug/L	97
47)	Tetrachloroethene	21.07	129	393202	38.95	ug/L	98
48)	1,3-Dichloropropane	21.11	76	435860	39.37	ug/L	95
49)	2-Hexanone	21.36	43	317400	45.03	ug/L	99
50)	Dibromochloromethane	21.65	129	458813	38.87	ug/L	99
51)	1,2-Dibromoethane	21.89	109	426778	39.15	ug/L	100
52)	Chlorobenzene	23.11	112	1229617	37.82	ug/L	98
53)	1,1,1,2-Tetrachloroethane	23.29	131	534496	39.16	ug/L	99
54)	Ethyl Benzene	23.37	91	2227008	37.91	ug/L	98
55)	m/p-Xylene	23.67	91	4194066	79.49	ug/L	99
56)	o-Xylene	24.65	91	2092321	38.80	ug/L	100
57)	Styrene	24.68	104	1226631	37.48	ug/L	95
58)	Bromoform	25.13	173	268696	38.95	ug/L	98
59)	Isopropylbenzene	25.59	105	2352139	39.51	ug/L	99
62)	1,4-trans-2-dichlorobutene	26.49	53	160897	37.81	ug/L	96
63)	Bromobenzene	26.33	156	660946	39.32	ug/L	98
64)	1,1,2,2-Tetrachloroethane	26.35	83	477288	39.70	ug/L	98
65)	1,2,3-Trichloropropane	26.44	75	686289	36.02	ug/L	94
66)	n-Propylbenzene	26.63	91	2692043	38.12	ug/L	99
67)	2-Chlorotoluene	26.84	126	632243	39.95	ug/L	99
68)	1,3,5-Trimethylbenzene	27.09	105	2183213	38.50	ug/L	99
69)	4-Chlorotoluene	27.12	91	2017425	38.48	ug/L	100
70)	tert-Butylbenzene	27.92	119	2139901	38.96	ug/L	99
71)	1,2,4-Trimethylbenzene	28.04	105	2061299	35.85	ug/L	99
72)	sec-Butylbenzene	28.47	105	2562219	38.83	ug/L #	96
73)	1,3-Dichlorbenzene	28.71	146	1102654	39.88	ug/L	98
74)	4-Isopropyltoluene	28.80	119	2215554	38.98	ug/L	97
75)	1,4-Dichlorobenzene	28.91	146	1006604	37.08	ug/L	97
76)	1,2-Dichlorobenzene	29.72	146	946163	39.51	ug/L	99
77)	n-Butylbenzene	29.71	91	2136695	39.85	ug/L	96
78)	1,2-Dibromo-3-chloropropan	31.31	75	182683	39.49	ug/L	96
79)	1,2,4-Trichlorobenzene	33.08	180	597182	38.54	ug/L	99
80)	Hexachlorobutadiene	33.48	225	551216	38.82	ug/L #	94
81)	Naphthalene	33.65	128	1402671	40.90	ug/L	99
82)	1,2,3-Trichlorobenzene	34.23	180	590953	38.70	ug/L	99

(#) = qualifier out of range (m) = manual integration
CCP0301.D 8260B-3.M Tue Mar 02 09:57:37 1999

Quantitation Report

Data File : c:\hpchem\1\data\CCP0301.D Vial: 0
Acq On : 1 MAR 1999 13:36 Operator:
Sample : Inst :
Misc : Multiplr: 1.00
MS Integration Params: rteint.p
Quant Time: Mar 1 14:13 1999 Quant Results File: 8260b-3.RES

Method : c:\sat132\quant\8260b-3.m (RTE Integrator)
Title : Saturn 132
Last Update : Fri Feb 26 15:11:05 1999
Response via : Initial Calibration



Quantitation Report

Data File : C:\HPCHEM\1\DATA\CDP0301.D Vial: 0
Acq On : 1 MAR 1999 14:59 Operator:
Sample : Inst :
Misc : Multiplr: 1.00
MS Integration Params: rteint.p
Quant Time: Mar 2 9:36 1999 Quant Results File: 8260B-3.RES

Quant Method : c:\sat132\quant\8260b-3.m (RTE Integrator)
Title : Saturn 132
Last Update : Fri Feb 26 15:11:05 1999
Response via : Initial Calibration
DataAcq Meth :
IS QA File : C:\HPCHEM\1\DATA\CCP0226.D (26 FEB 1999 12:14)

Internal Standards	R.T.	QIon	Response	Conc	Units		Dev(Min) Rcv(Ar)
1) IS: Flourobenzene	16.12	96	1309609	40.00	ug/L		-0.02 98.80%
43) IS: d5-Chlorobenzene	23.03	82	708424	40.00	ug/L		0.00 93.30%
61) IS: d4-1,4-Dichlorobenzene	28.84	152	505996	40.00	ug/L		-0.01 85.26%
System Monitoring Compounds							
28) SUR: Dibromofluoromethane	14.69	113	628999	58.97	ug/L		-0.04
Spiked Amount 40.000			Recovery =	147.43%			
34) SUR: 1,2-Dichloroethane-d4	15.38	65	510893	40.93	ug/L		-0.03
Spiked Amount 40.000			Recovery =	102.33%			
42) SUR: d8-Toluene	19.63	98	2474311	61.43	ug/L		-0.02
Spiked Amount 40.000			Recovery =	153.58%			
60) Sur: 4-Bromofluorobenzene	25.95	95	1196107	62.06	ug/L		-0.0[illegible]
Spiked Amount 40.000			Recovery =	155.15%			
Target Compounds							Qvalue
2) Dichlorodifluoromethane	2.97	85	669944	113.55	ug/L		100
3) Chloromethane	3.88	50	528387	110.49	ug/L		98
4) Vinyl Chloride	4.44	62	748901	113.67	ug/L		98
5) Bromomethane	5.88	94	900050	126.69	ug/L		99
6) Chloroethane	6.37	45	76976	110.66	ug/L	#	70
7) Trichlorofluoromethane	7.40	101	1870151	117.00	ug/L		99
8) Ethyl Ether	8.62	45	533607	116.77	ug/L		97
9) Acrolein	8.90	55	63817	124.90	ug/L		93
10) 1,1-Dichloroethene	9.06	96	1010875	116.19	ug/L		99
11) Trichlorotrifluoroethane	9.25	101	1444849	115.39	ug/L	#	79
12) Iodomethane	9.33	142	2823331	137.53	ug/L		99
13) Carbon Disulfide	9.40	76	2116244	114.26	ug/L		100
14) Acetone	9.44	43	407524	118.47	ug/L	#	98
15) Isopropanol	10.30	45	30687	112.87	ug/L	#	92
16) Methylene Chloride	10.53	49	2271642	114.55	ug/L		98
17) Acrylonitrile	11.24	52	477945	114.75	ug/L	#	90
18) trans-1,2-Dichloroethene	11.22	96	1156453	124.30	ug/L		93
19) Methyl(tert)Butyl Ether	11.33	73	3353266	118.26	ug/L		99
20) 1,1-Dichloroethane	12.23	63	1665017	119.42	ug/L		99
21) Vinyl Acetate	12.53	43	1936626m	131.35	ug/L		98
22) 2,2-Dichloropropane	13.53	77	2108970	116.56	ug/L		97
23) cis-1,2-Dichloroethene	13.58	96	1203024	115.68	ug/L		97
24) Methyl Ethyl Ketone	13.72	43	534524	126.01	ug/L		[illegible]
25) Bromochloromethane	14.11	49	1551093	121.49	ug/L		99
26) Chloroform	14.34	83	2221432	111.76	ug/L		98
27) 1,1,1-Trichloroethane	14.67	97	2604561	117.12	ug/L		100

29)	Carbon Tetrachloride	15.02	117	1863391	113.05	ug/L	100
30)	1,1-Dichloropropene	15.05	75	1725494	110.29	ug/L	98
31)	Benzene	15.49	78	4799590	114.90	ug/L	98
32)	1,2-Dichloroethane	15.54	62	1903936	117.07	ug/L	99
33)	Heptane	16.16	41	2177875	117.50	ug/L	99
35)	Trichloroethene	16.92	130	1255936	115.01	ug/L	96
36)	1,2-Dichloropropane	17.39	63	839828	114.03	ug/L	97
37)	Dibromomethane	17.66	93	969398	114.00	ug/L	99
38)	Bromodichloromethane	18.04	83	1668206	107.62	ug/L	97
39)	2-Chloroethyl Vinyl Ether	18.74	63	790013	118.38	ug/L #	99
40)	cis-1,3-Dichloropropene	19.05	75	1997325	113.78	ug/L	99
41)	4-Methyl-2-Pentanone	19.42	43	1145562	116.09	ug/L	98
44)	Toluene	19.78	91	6106305	118.03	ug/L	99
45)	trans-1,3-Dichloropropene	20.32	75	1870573	121.81	ug/L	99
46)	1,1,2-Trichloroethane	20.73	83	829455	123.16	ug/L	98
47)	Tetrachloroethene	21.06	129	1040771	106.56	ug/L	97
48)	1,3-Dichloropropane	21.11	76	1361312	127.08	ug/L #	92
49)	2-Hexanone	21.33	43	909248	133.32	ug/L	99
50)	Dibromochloromethane	21.64	129	1386397	121.40	ug/L	97
51)	1,2-Dibromoethane	21.88	109	1266394	120.06	ug/L	98
52)	Chlorobenzene	23.08	112	3699007	117.59	ug/L	99
53)	1,1,1,2-Tetrachloroethane	23.29	131	1547355	117.17	ug/L	99
54)	Ethyl Benzene	23.37	91	6578219	115.73	ug/L	99
55)	m/p-Xylene	23.67	91	11872274	232.54	ug/L	97
56)	o-Xylene	24.64	91	6063457	116.22	ug/L	98
57)	Styrene	24.68	104	3733786	117.92	ug/L	95
58)	Bromoform	25.11	173	801632	120.10	ug/L	98
59)	Isopropylbenzene	25.56	105	6940370	120.49	ug/L	97
62)	1,4-trans-2-dichlorobutene	26.48	53	504126	124.27	ug/L	99
63)	Bromobenzene	26.32	156	1957217	122.12	ug/L	83
64)	1,1,2,2-Tetrachloroethane	26.33	83	1402472	122.37	ug/L	99
65)	1,2,3-Trichloropropane	26.43	75	2129350m	117.21	ug/L	99
66)	n-Propylbenzene	26.61	91	7861026	116.75	ug/L	98
67)	2-Chlorotoluene	26.83	126	1832034	121.42	ug/L	97
68)	1,3,5-Trimethylbenzene	27.08	105	6311880	116.73	ug/L	99
69)	4-Chlorotoluene	27.10	91	5976603	119.57	ug/L	97
70)	tert-Butylbenzene	27.90	119	5987106	114.33	ug/L	97
71)	1,2,4-Trimethylbenzene	28.03	105	6350548	115.85	ug/L	99
72)	sec-Butylbenzene	28.45	105	7464241	118.65	ug/L #	98
73)	1,3-Dichlorbenzene	28.69	146	3100977	117.62	ug/L	99
74)	4-Isopropyltoluene	28.78	119	6326453	116.75	ug/L	97
75)	1,4-Dichlorobenzene	28.89	146	3091334	119.44	ug/L	97
76)	1,2-Dichlorobenzene	29.72	146	2501555	109.56	ug/L	97
77)	n-Butylbenzene	29.69	91	6027417	117.91	ug/L #	94
78)	1,2-Dibromo-3-chloropropan	31.28	75	551794	125.09	ug/L	96
79)	1,2,4-Trichlorobenzene	33.07	180	1748934	118.40	ug/L	98
80)	Hexachlorobutadiene	33.47	225	1578691	116.62	ug/L	99
81)	Naphthalene	33.64	128	4103799	125.50	ug/L	100
82)	1,2,3-Trichlorobenzene	34.20	180	1659765	113.99	ug/L	99

(#) = qualifier out of range (m) = manual integration
CDP0301.D 8260B-3.M Tue Mar 02 09:59:17 1999

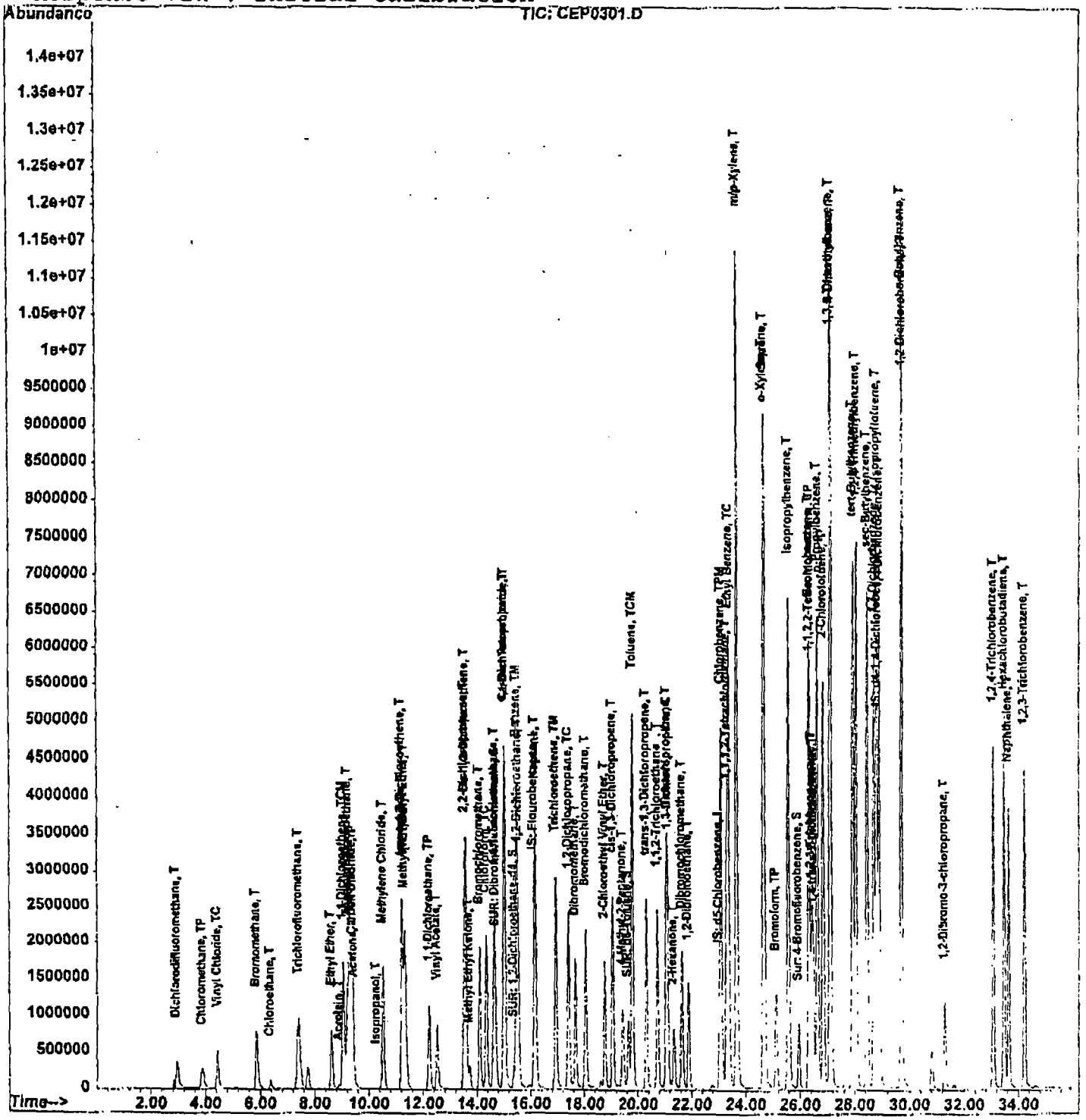
29)	Carbon Tetrachloride	15.04	117	3553200	220.42	ug/L	
30)	1,1-Dichloropropene	15.04	75	3217474	216.05	ug/L	99
31)	Benzene	15.50	78	9089595	228.59	ug/L	96
32)	1,2-Dichloroethane	15.55	62	3490465	225.47	ug/L	95
33)	Heptane	16.17	41	4094205	232.05	ug/L	98
35)	Trichloroethene	16.94	130	2297064	220.97	ug/L	98
36)	1,2-Dichloropropane	17.42	63	1501637	214.19	ug/L	9[illegible]
37)	Dibromomethane	17.68	93	1740020	214.97	ug/L	99
38)	Bromodichloromethane	18.06	83	3287118	222.78	ug/L	98
39)	2-Chloroethyl Vinyl Ether	18.77	63	1463806	230.42	ug/L #	98
40)	cis-1,3-Dichloropropene	19.06	75	3912089	234.12	ug/L	99
41)	4-Methyl-2-Pentanone	19.44	43	2050691	218.31	ug/L	98
44)	Toluene	19.80	91	11281945	223.24	ug/L	99
45)	trans-1,3-Dichloropropene	20.33	75	3438231	229.20	ug/L	100
46)	1,1,2-Trichloroethane	20.74	83	1385993	210.67	ug/L	96
47)	Tetrachloroethene	21.08	129	2091317	219.19	ug/L	96
48)	1,3-Dichloropropane	21.13	76	2275818	217.48	ug/L	94
49)	2-Hexanone	21.35	43	1659120	249.03	ug/L	98
50)	Dibromochloromethane	21.66	129	2568663	230.25	ug/L	99
51)	1,2-Dibromoethane	21.90	109	2356532	228.70	ug/L	97
52)	Chlorobenzene	23.11	112	6982211	227.21	ug/L	97
53)	1,1,1,2-Tetrachloroethane	23.31	131	2926492	226.86	ug/L	100
54)	Ethyl Benzene	23.40	91	12662048	228.04	ug/L	96
55)	m/p-Xylene	23.69	91	20625172	413.55	ug/L	97
56)	o-Xylene	24.66	91	10759791	211.12	ug/L	95
57)	Styrene	24.69	104	7205076	232.95	ug/L #	82
58)	Bromoform	25.13	173	1501860	230.34	ug/L	94
59)	Isopropylbenzene	25.58	105	13125309	233.25	ug/L	98
62)	1,4-trans-2-dichlorobutene	26.51	53	973147	259.08	ug/L	98
63)	Bromobenzene	26.34	156	3247025	218.81	ug/L	85
64)	1,1,2,2-Tetrachloroethane	26.36	83	2563939	241.61	ug/L	97
65)	1,2,3-Trichloropropane	26.45	75	3675081m	218.49	ug/L	99
66)	n-Propylbenzene	26.65	91	14533242	233.12	ug/L	9[illegible]
67)	2-Chlorotoluene	26.84	126	3365099	240.87	ug/L	[illegible]
68)	1,3,5-Trimethylbenzene	27.10	105	11757742	234.85	ug/L	97
69)	4-Chlorotoluene	27.13	91	10825620	233.92	ug/L	96
70)	tert-Butylbenzene	27.94	119	11377552	234.64	ug/L	99
71)	1,2,4-Trimethylbenzene	28.06	105	11645228	229.43	ug/L	99
72)	sec-Butylbenzene	28.46	105	13782543	236.61	ug/L	100
73)	1,3-Dichlorbenzene	28.71	146	5910380	242.12	ug/L	96
74)	4-Isopropyltoluene	28.81	119	12063503	240.45	ug/L #	90
75)	1,4-Dichlorobenzene	28.91	146	5417604	226.06	ug/L	98
76)	1,2-Dichlorobenzene	29.75	146	4362160	206.34	ug/L	99
77)	n-Butylbenzene	29.71	91	11438787	241.68	ug/L #	89
78)	1,2-Dibromo-3-chloropropan	31.30	75	966551	236.66	ug/L	98
79)	1,2,4-Trichlorobenzene	33.08	180	3129867	228.84	ug/L	97
80)	Hexachlorobutadiene	33.49	225	2793291	222.85	ug/L	98
81)	Naphthalene	33.64	128	6845171	226.09	ug/L	100
82)	1,2,3-Trichlorobenzene	34.23	180	3121037	231.51	ug/L	98

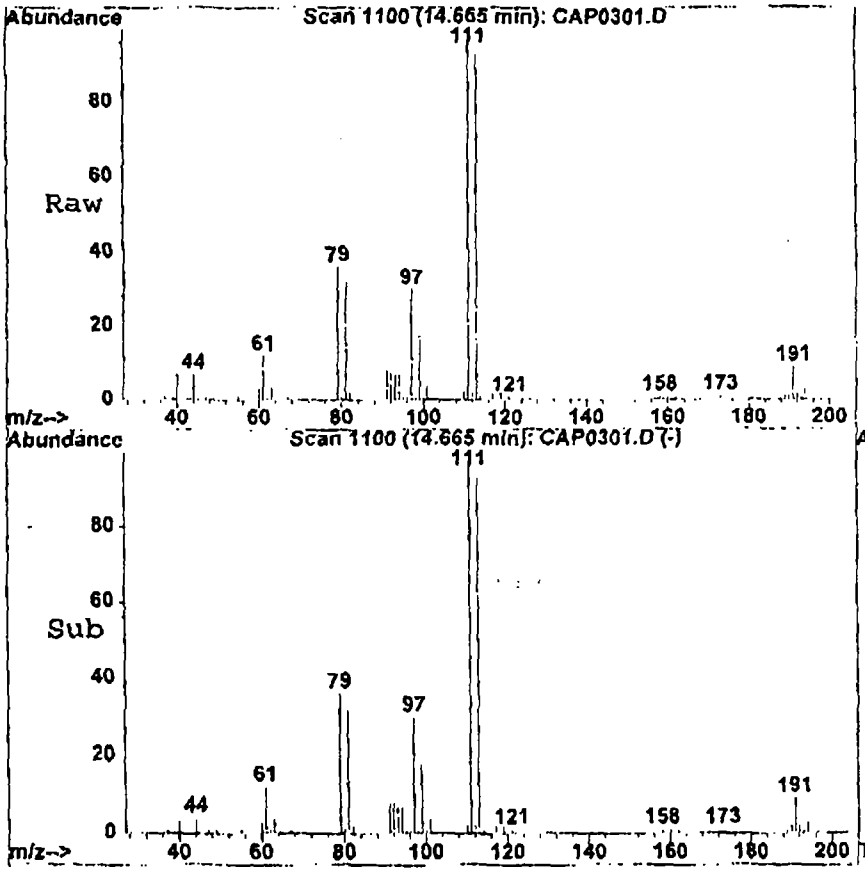
(#) = qualifier out of range (m) = manual integration
CEP.0301.D 8260B-3.M Tue Mar 02 10:00:59 1999

Quantitation Report

Data File : c:\hpchem\1\data\CEP0301.D Vial: 0
Acq On : 1 MAR 1999 17:05 Operator:
Sample : Inst :
Misc : Multiplr: 1.00
MS Integration Params: rteint.p
Quant Time: Mar 1 17:43 1999 Quant Results File: 8260b-3.RES

Method : c:\sat132\quant\8260b-3.m (RTE Integrator)
Title : Saturn 132
Last Update : Fri Feb 26 15:11:05 1999
Response via : Initial Calibration

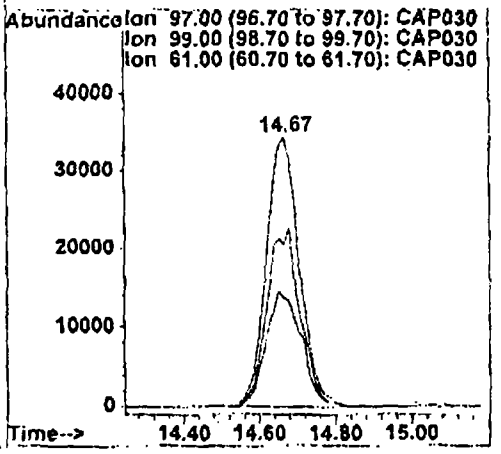


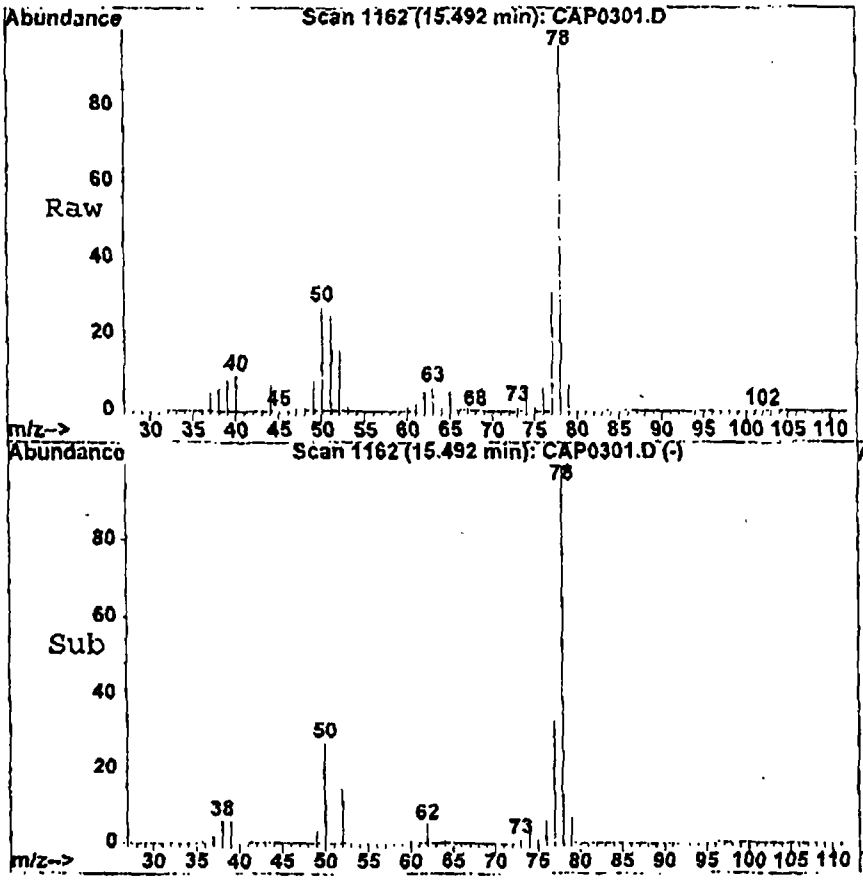


#27
1,1,1-Trichloroethane
Concen: 9.50 ug/L
RT: 14.67 min Scan# 1100
Delta R.T. -0.04 min
Lab File: CAP0301.D
Acq: 1 MAR 1999 12:12

Tgt Ion: 97 Resp: 208386

Ion	Ratio	Lower	Upper
97	100		
99	66.0	52.6	79.0
61	46.0	38.3	57.5

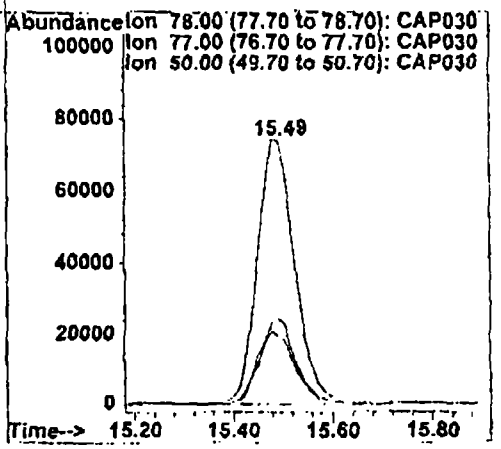


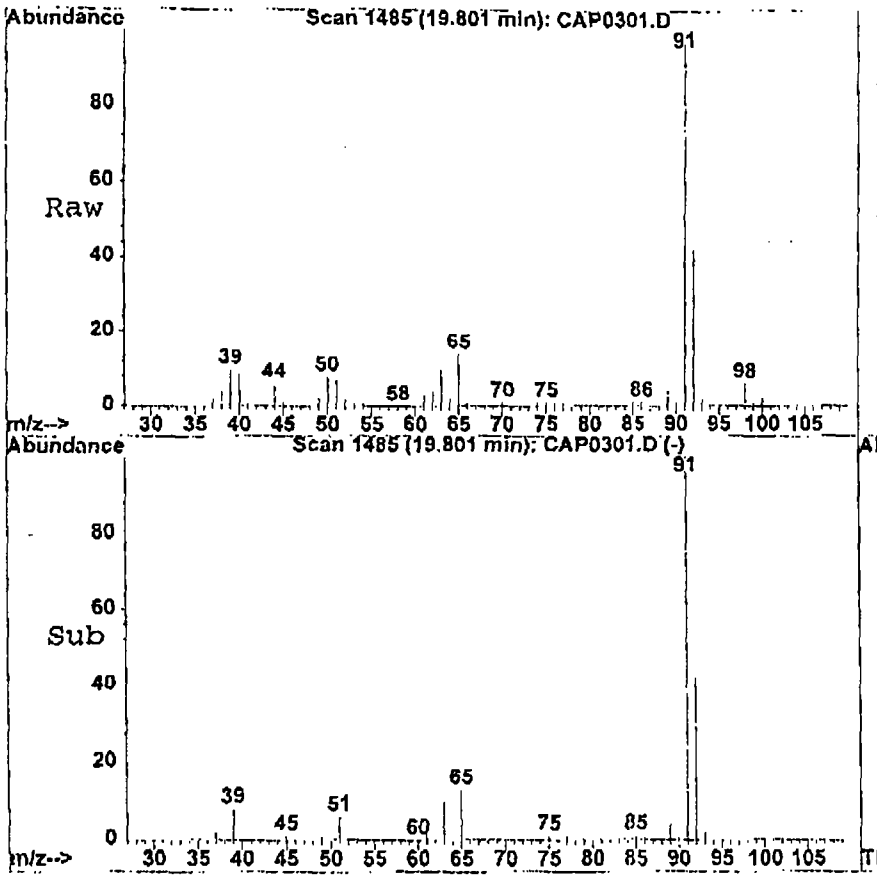


#31
Benzene
Concen: 9.69 ug/L
RT: 15.49 min Scan# 1162
Delta R.T. -0.03 min
Lab File: CAP0301.D
Acq: 1 MAR 1999 12:12

Tgt Ion: 78 Resp: 399271

Ion	Ratio	Lower	Upper
78	100		
77	32.4	25.2	37.8
50	28.6	20.8	31.2

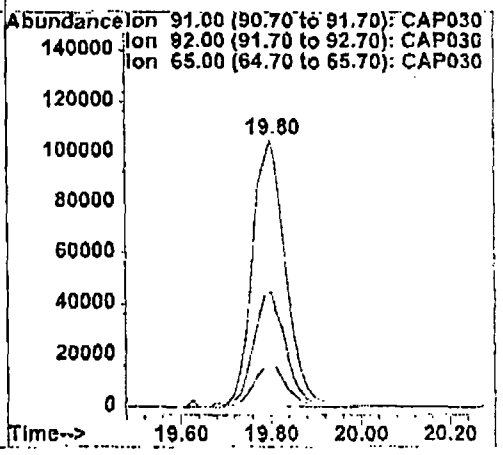


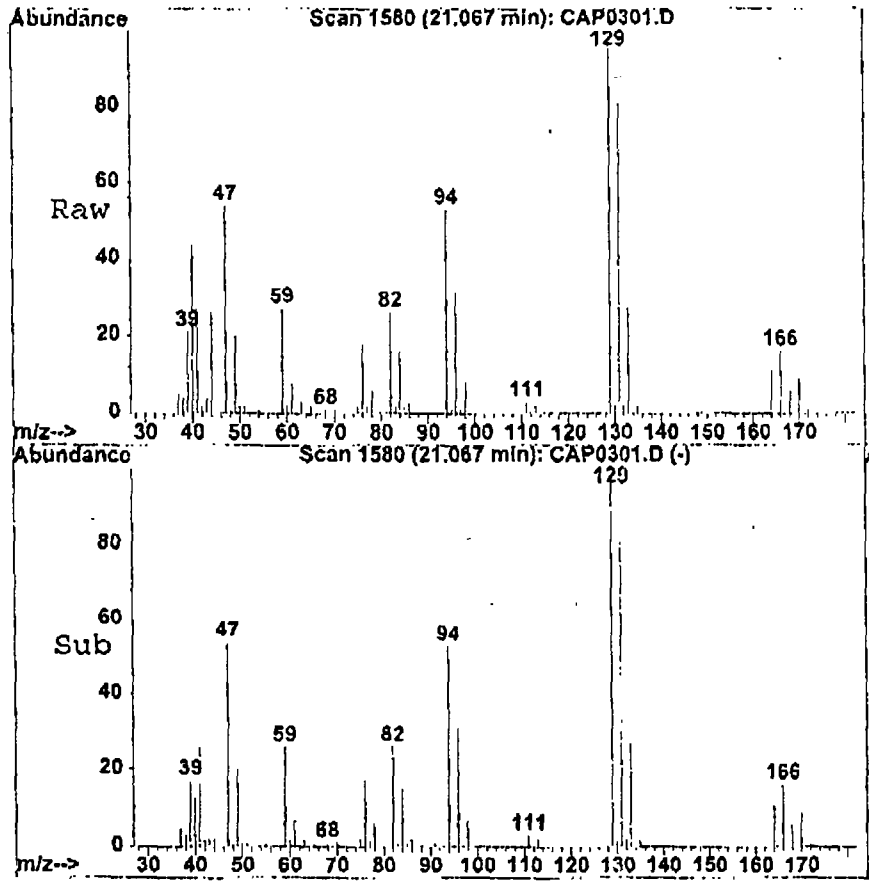


#44
Toluene
Concen: 9.77 ug/L
RT: 19.80 min Scan# 1485
Delta R.T. 0.00 min
Lab File: CAP0301.D
Acq: 1 MAR 1999 12:12

Tgt Ion: 91 Resp: 535150

Ion	Ratio	Lower	Upper
91	100		
92	41.8	32.9	49.3
65	14.8	11.8	17.8

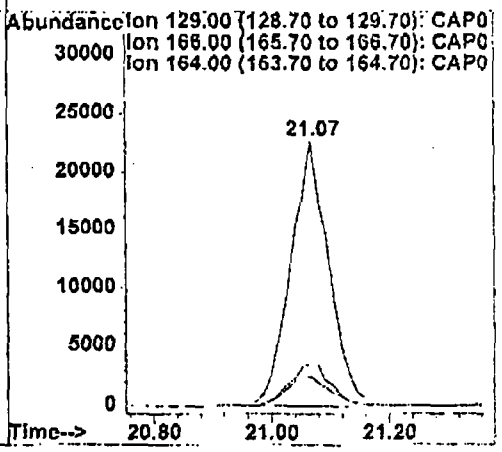


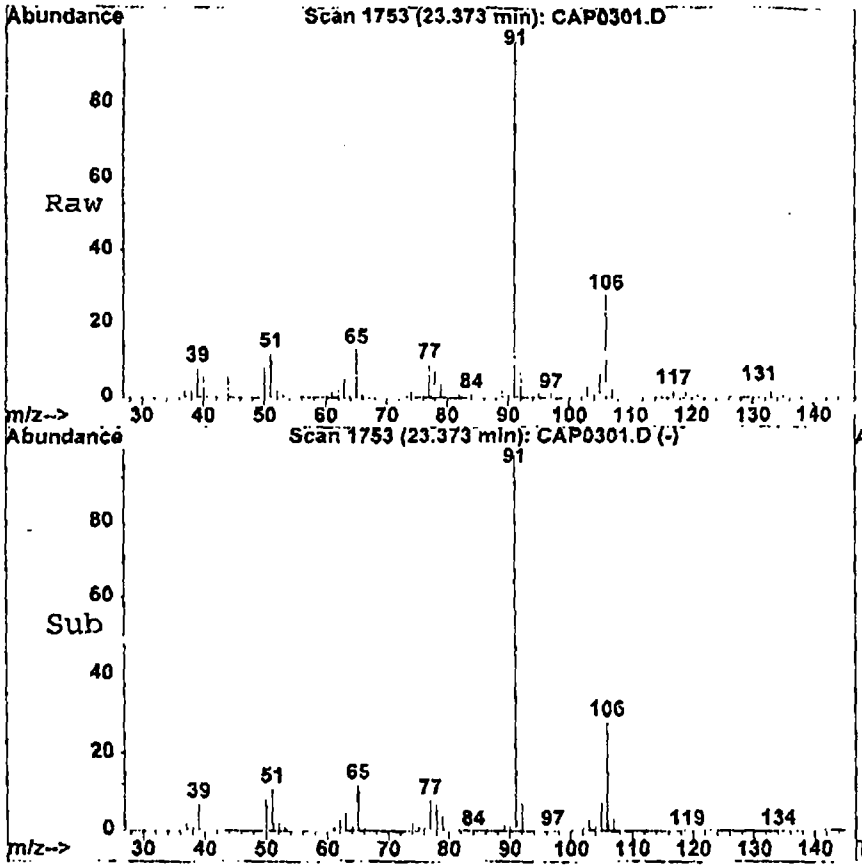


#47
Tetrachloroethene
Concen: 9.78 ug/L
RT: 21.07 min Scan# 1580
Delta R.T. 0.00 min
Lab File: CAP0301.D
Acq: 1 MAR 1999 12:12

Tgt Ion:129 Resp: 101101

Ion	Ratio	Lower	Upper
129	100		
166	17.0	14.2	21.4
164	12.2	11.5	17.3

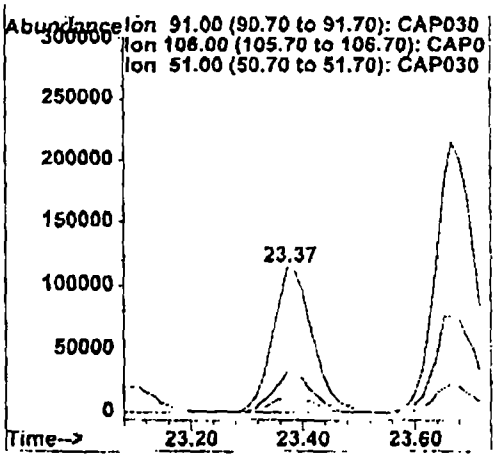


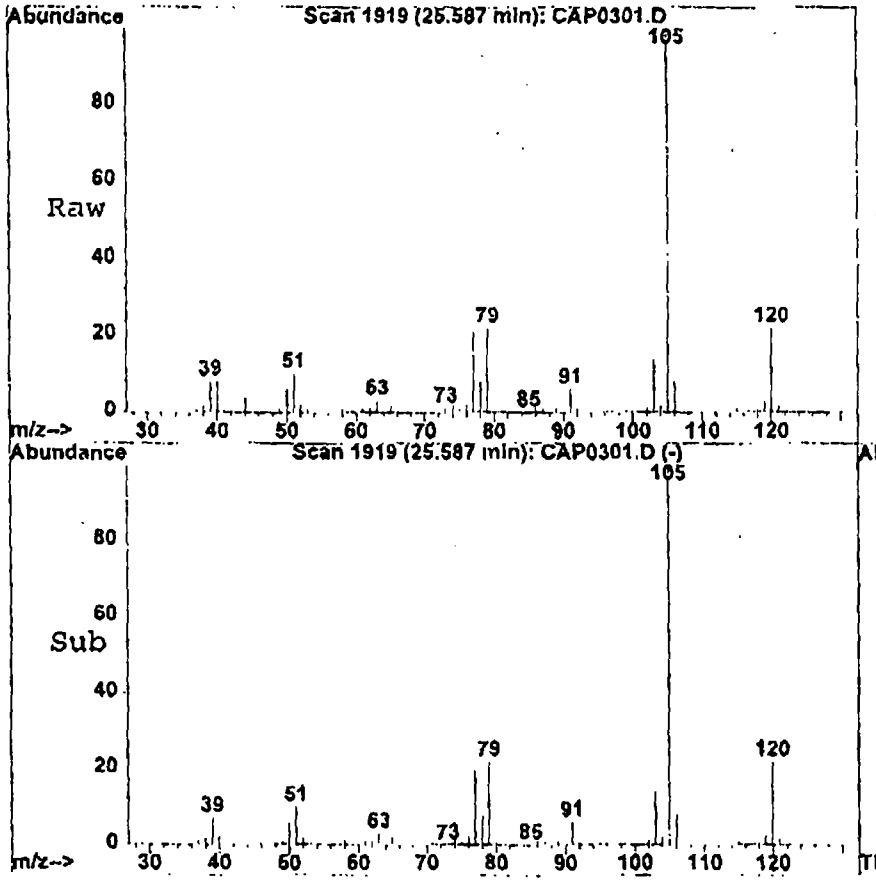


#54
Ethyl Benzene
Concen: 9.34 ug/L
RT: 23.37 min Scan# 1753
Delta R.T. -0.00 min
Lab File: CAP0301.D
Acq: 1 MAR 1999 12:12

Tgt Ion: 91 Resp: 562128

Ion	Ratio	Lower	Upper
91	100		
106	27.2	21.4	32.2
51	12.0	8.6	12.8

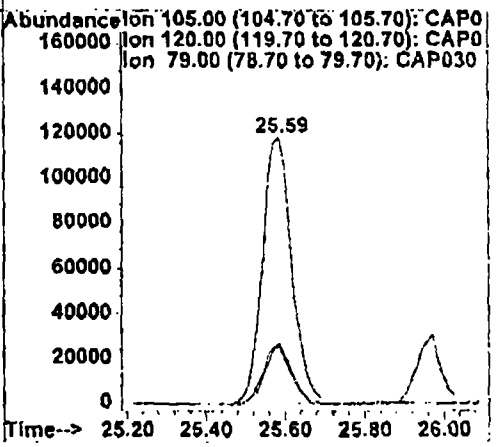


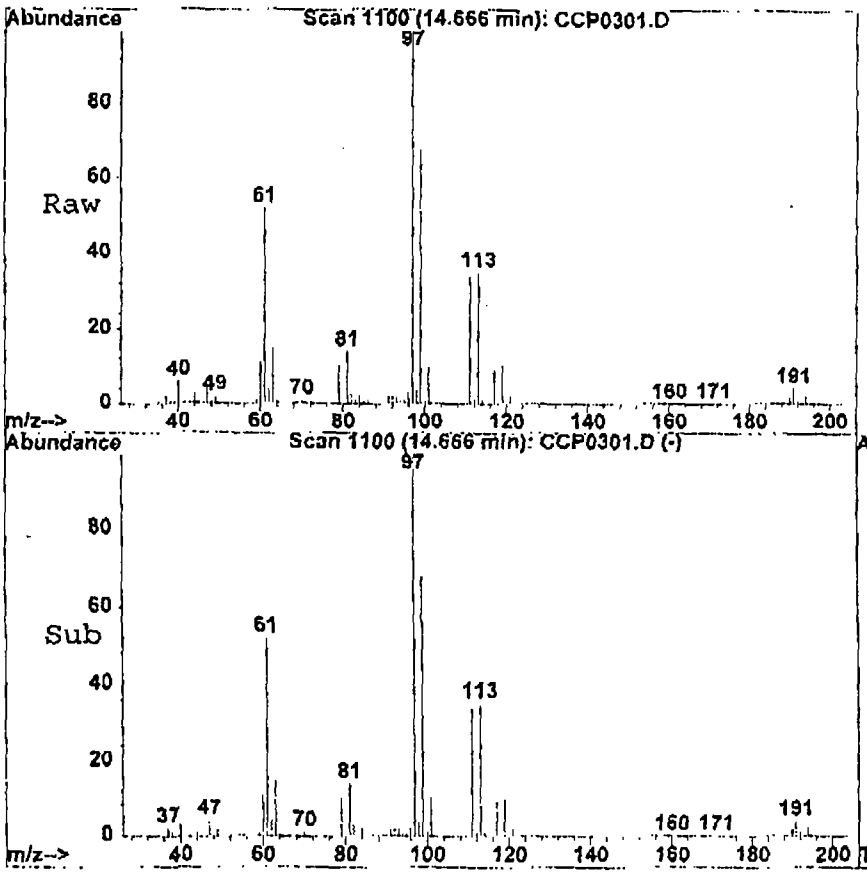


#59
Isopropylbenzene
Concen: 9.93 ug/L
RT: 25.59 min Scan# 19
Delta R.T. 0.00 min
Lab File: CAP0301.D
Acq: 1 MAR 1999 12:12

Tgt Ion:105 Resp: 605632

Ion	Ratio	Lower	Upper
105	100		
120	19.3	15.1	22.7
79	21.6	16.6	24.8

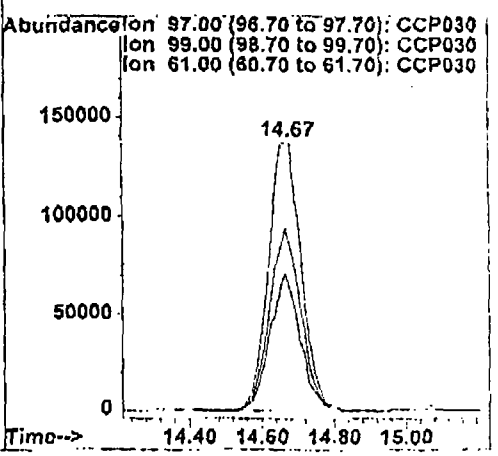


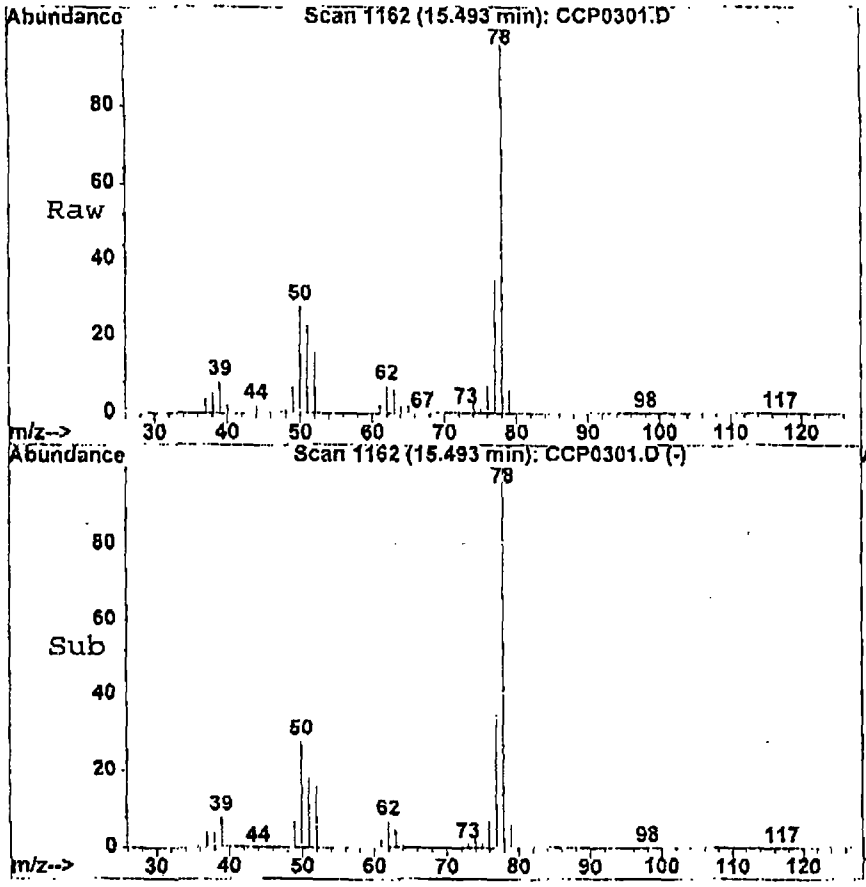


#27
1,1,1-Trichloroethane
Concen: 39.69 ug/L
RT: 14.67 min Scan# 1100
Delta R.T. -0.04 min
Lab File: CCP0301.D
Acq: 1 MAR 1999 13:36

Tgt Ion: 97 Resp: 867861

Ion	Ratio	Lower	Upper
97	100		
99	65.0	52.6	79.0
61	47.6	38.3	57.5

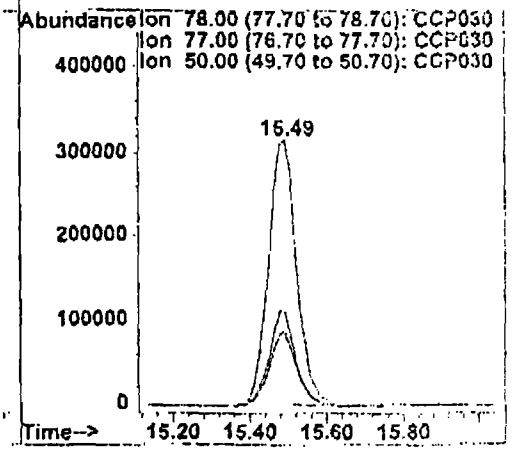


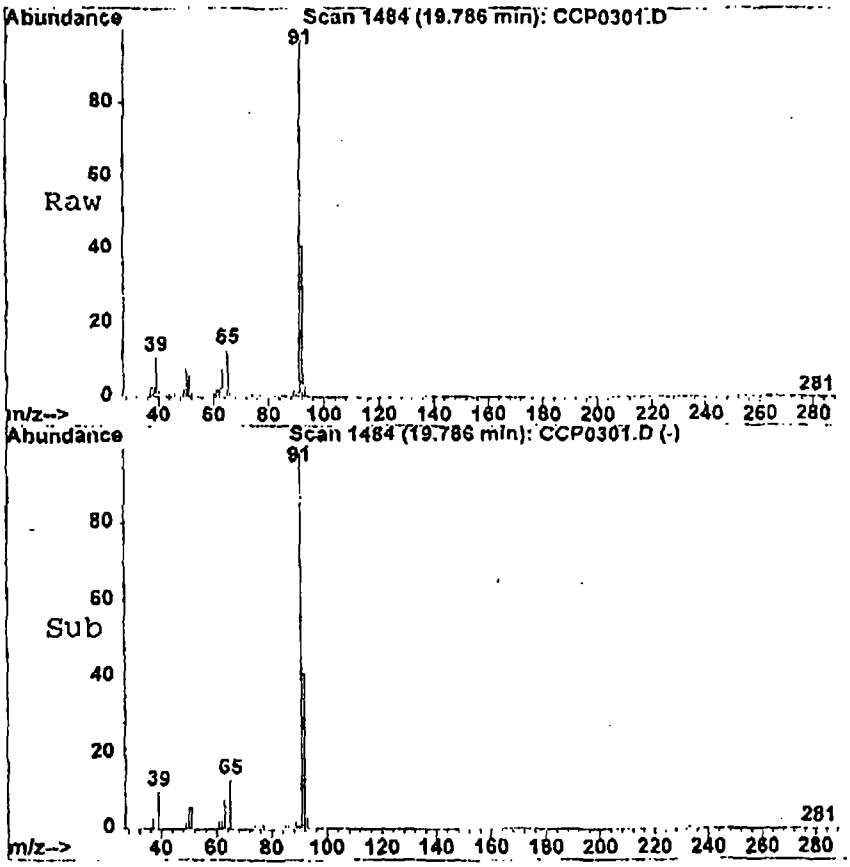


#31
Benzene
Concen: 38.89 ug/L
RT: 15.49 min Scan# 1162
Delta R.T. -0.03 min
Lab File: CCP0301.D
Acq: 1 MAR 1999 13:36

Tgt Ion: 78 Resp: 1597216

Ion	Ratio	Lower	Upper
78	100		
77	32.8	25.2	37.8
50	27.4	20.8	31.2

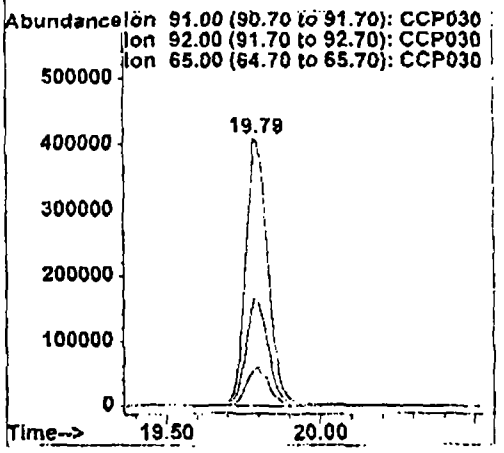


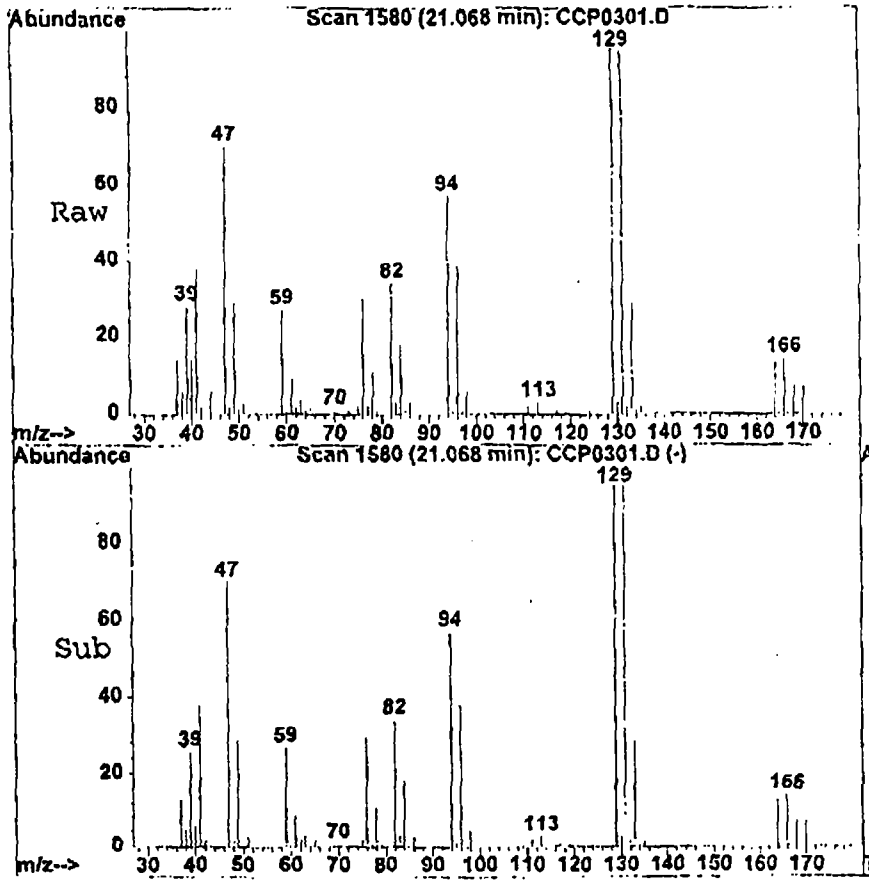


#44
Toluene
Concen: 37.49 ug/L
RT: 19.79 min Scan# 1484
Delta R.T. -0.01 min
Lab File: CCP0301.D
Acq: 1 MAR 1999 13:36

Tgt Ion: 91 Resp: 2004465

Ion	Ratio	Lower	Upper
91	100		
92	40.8	32.9	49.3
65	14.2	11.8	17.8

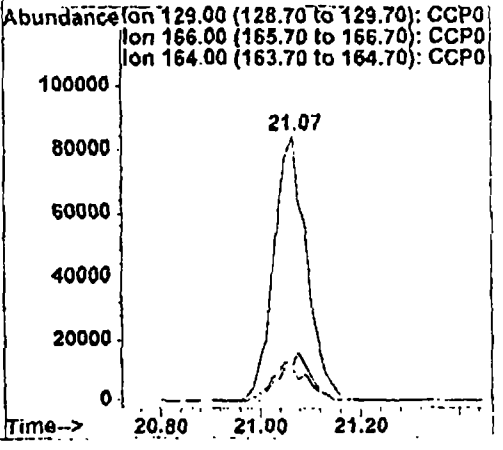


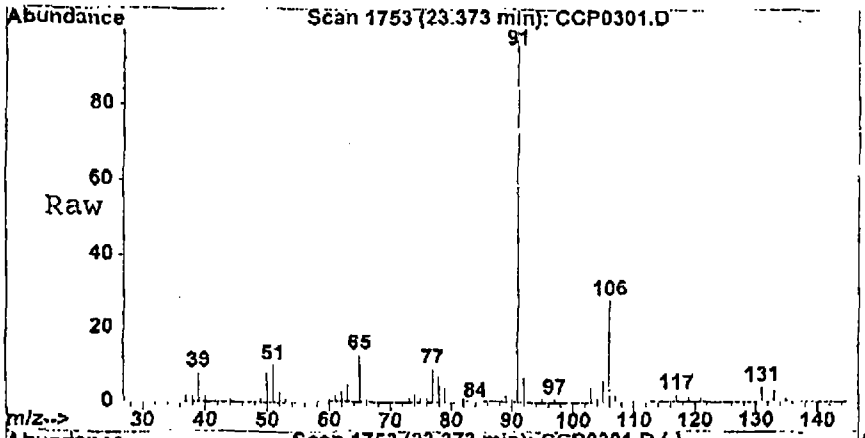


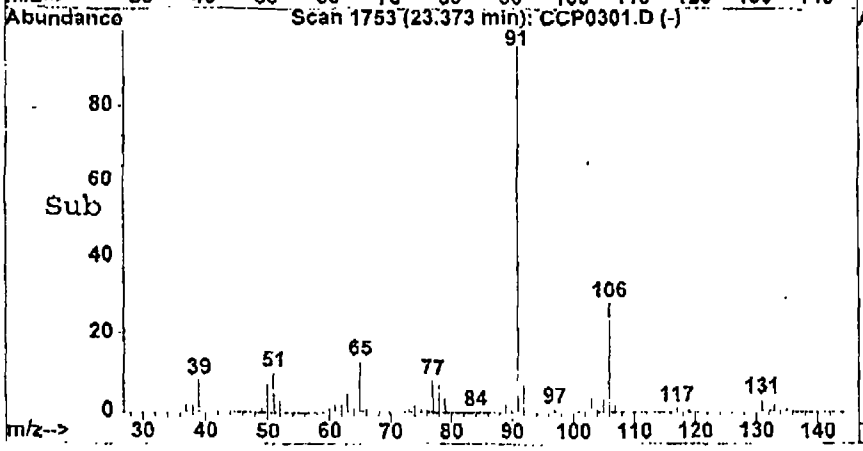
#47
Tetrachloroethene
Concen: 38.95 ug/L
RT: 21.07 min Scan# 158
Delta R.T. 0.00 min
Lab File: CCP0301.D
Acq: 1 MAR 1999 13:36

Tgt Ion:129 Resp: 393202

Ion	Ratio	Lower	Upper
129	100		
166	18.4	14.2	21.4
164	13.4	11.5	17.3



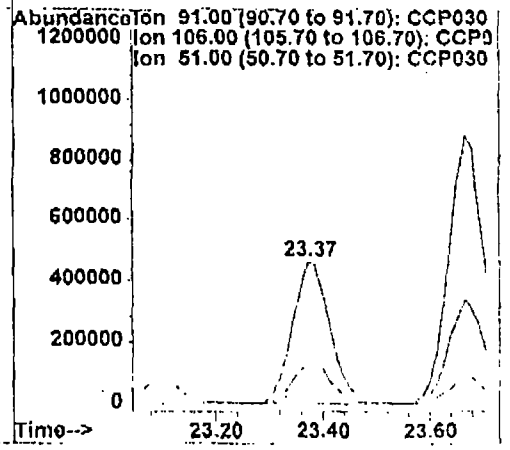


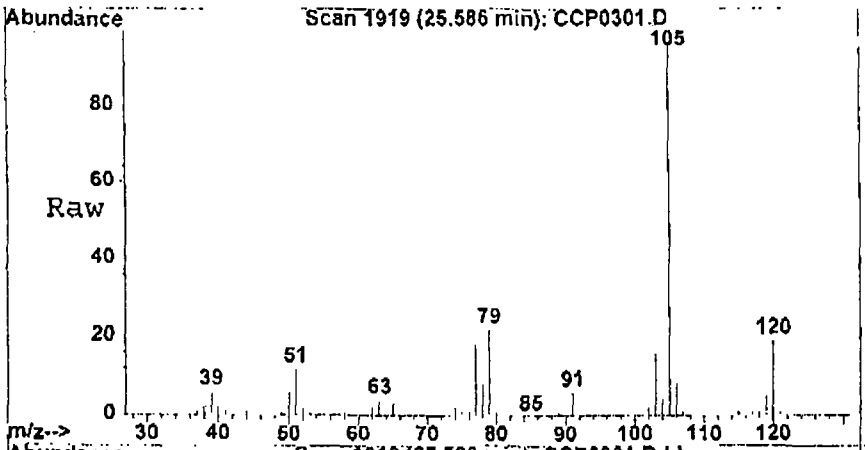


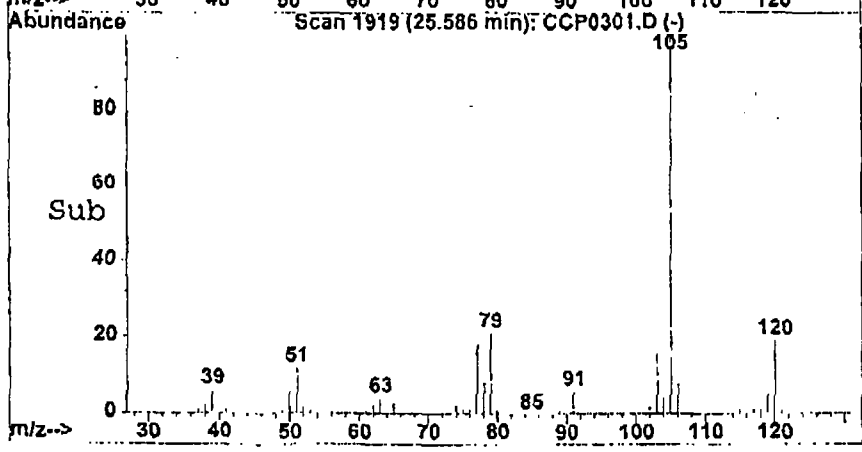
#54
Ethyl Benzene
Concen: 37.91 ug/L
RT: 23.37 min Scan# 1753
Delta R.T. -0.00 min
Lab File: CCP0301.D
Acq: 1 MAR 1999 13:36

Tgt Ion: 91 Resp: 2227008

Ion	Ratio	Lower	Upper
91	100		
106	28.2	21.4	32.2
51	11.2	8.6	12.8



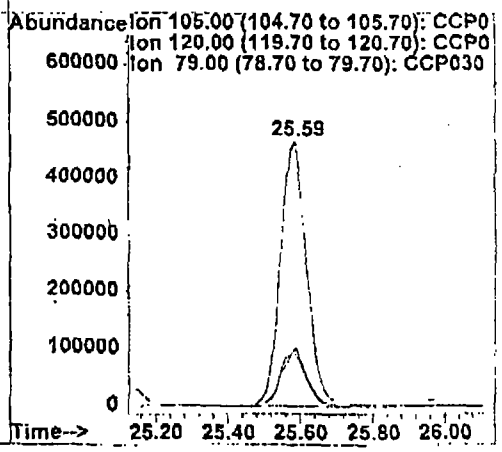




#59
Isopropylbenzene
Concen: 39.51 ug/L
RT: 25.59 min Scan# 1919
Delta R.T. 0.00 min
Lab File: CCP0301.D
Acq: 1 MAR 1999 13:36

Tgt Ion:105 Resp: 2352139

Ion	Ratio	Lower	Upper
105	100		
120	19.3	15.1	22.7
79	21.1	16.6	24.8



TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

VOLATILE ORGANIC LABORATORY CORRECTIVE ACTIONS

APPROVALS:

Organic Manager: _____ Date: 11/5/97
Jeff Glaser

QA Manager: _____ Date: 11/5/97
Rick D. Wilburn

Laboratory Manager: _____ Date: 11/5/97
Douglas E. Kriscunas

Procedure Number: GR-03-124
Revision Number: 2.0

Date Initiated: 3/22/95
Effective Date: 11/5/97

Date Revised: 11/5/97
Pages Revised: All

By: Jeff Glaser

Total Number of Pages: 11

1.0 SUMMARY OF PROCEDURE

1.1 Sample results must conform to specified method quality control criteria. Every effort will be made to produce results that conform with those criteria. When this is not possible and an out of control data point occurs, it is important for the analyst to recognize the problem, then qualify and narrate the data correctly.

1.2 This SOP provides guidelines for corrective actions regarding out of control sample data points. It provides instructions on both the procedure to follow when a data point is out of control, and how to narrate and qualify the data when necessary. It is the responsibility of the analyst to inform the project chemist when a situation arises where he feels it is important to notify the client now, rather than when they receive the data package. The analyst must to do everything in his power to prevent the qualification of data when possible. Re-injections, re-extractions, and alternate cleanup methods will all be employed when practical to do so.

1.3 Non-sample related quality control deviations such as out of control tunes or continuing calibrations are not covered in this SOP. Those items are covered in the appropriate analytical SOP. The more common instances where a sample will require qualification have been covered in this SOP. Analyst experience will govern the correct course of action in other situations.

2.0 DETAILED PROCEDURE

2.1 Surrogate Failure

2.1.1 When a surrogate fails recovery criteria, deciding on how to proceed is influenced by a number of factors. Is re-analysis required (ie. should the sample be re-run or a new methanol extract prepared)? Does the data require qualification, and if so how should the data be qualified? Analyst experience is critical in determining the correct course of action. Surrogate failures generally fall into three categories: sample matrix interference, preparation error, or instrument failure. The course of action taken is specific to those three categories, and by the following general rules.

a) If any surrogate has a percent recovery <10%, all data and detection limits for the corresponding fraction will be narrated and qualified as unusable. (See LIMS qualifier #17 and 18).

b) If any surrogate has a percent recovery of ≥10%, but the percent recovery is less than the laboratory generated recovery lower control limit, all positive hits and detection limits will be narrated and qualified as estimated for that fraction. All non-detectable results will be qualified as approximate. (See LIMS qualifier # 16).

c) If any surrogate has a percent recovery that exceeds the laboratory generated recovery upper control limit, all positive hits for that fraction will be qualified and

narrated as being estimated. No qualification is required on detection limits or on non-detectable results. (See LIMS qualifier # 15).

d) For all volatile water samples, non-diluted soil samples, and soil samples with less than a 1:500 dilution, surrogate recoveries will be calculated and reported, and data will be narrated and qualified when necessary. Surrogate recoveries will not be calculated, reported, or used to qualify data on soil samples run at a dilution of >1:500.

e) Surrogate recoveries outside of the laboratory generated windows will not be used to qualify quality control samples with acceptable spike compound recoveries. However, if a matrix spike or matrix spike duplicate has acceptable surrogate recoveries, and the non-spiked version of the sample has surrogate recoveries outside of the acceptable windows, the data will be narrated and qualified, or appropriate action will be taken according to rules outlined elsewhere in this section.

2.1.1.1 Sample Matrix

2.1.1.1.1 Surrogate failure due to sample matrix problems will be suspected when one or more of the following occur:

a) poor chromatography
b) raised baseline
c) only one or two surrogates are out of control
d) co-elution of the surrogate with another peak
e) recovery of all surrogates are low due to sample foaming while being purged.

If sample matrix is suspected to be the cause of a failed surrogate recovery and no dilution is required, the sample will not be re-run and the data will be narrated and qualified as estimated. If a dilution is required, the surrogates from the diluted run will be used to decide if qualifications are necessary. (See LIMS qualifier # 19).

2.1.1.1.2 If sample matrix is not suspected to be the cause of a failed surrogate recovery, then the sample should be re-run at the same preparation as the initial run, unless a dilution due to high analyte concentration is required. If a dilution is necessary, then the sample should be re-run at the least possible dilution. If a dilution >1:500 on a soil sample is necessary, surrogate results will not be reported and no data will be qualified. If a water sample was diluted or a soil sample was diluted ≤1:500, the surrogates from the diluted run only will be used and reported.

2.1.1.1.3 If a soil sample was run at a dilution of >1:500, surrogate recoveries will not be used in deciding if data should be qualified.

2.1.1.2 Preparation Error

2.1.1.2.1 If surrogates are out of the laboratory generated recovery windows, preparation error will be suspected if:

a) the analysis of samples run before and after the sample in question had acceptable surrogate recoveries
b) the internal standard areas are also unacceptable/mis-injection is assumed (with manual addition of standards)
c) base line sensitivity appears normal

2.1.1.2.2 If the sample is within hold time it will automatically be re-run or re-prepared. If the re-analysis works, the first run will not be used and no data will be qualified. If the re-analysis shows similar results, matrix interferences will be assumed and the data for that fraction will be narrated and qualified as estimated (see Section 2.1.1.1). The initial run will be the run that is reported. If the sample is out of its' hold time, the project chemist will be consulted and the analyst will proceed as instructed, by either qualifying the data for that fraction as estimated based on poor surrogate recoveries, or by having the sample re-analyzed. If the re-analysis works, the data will be narrated and qualified because it was re-analyzed outside of the hold time. (See LIMS qualifier # 1).

2.1.1.3 Instrument Failure

2.1.1.3.1 If surrogates are out of the laboratory generated recovery windows mis-injection or instrument failure will be suspected if:

a) other runs produced inconsistent results
b) the internal standard areas are not acceptable
c) base line sensitivity looks low
d) previously injected samples had poor matrixes which may have affected surrogate recoveries of the sample in question

2.1.1.3.2 If instrument failure is assumed the sample will automatically be re-run. If the re-run works the first run will not be used and no data will be qualified. If the re-run does not work matrix interferences will be assumed and the analyst will proceed according to Section 2.1.1.1 or 2.1.1.2 as appropriate.

2.1.2 Surrogates will also fail when the sample has been mis-spiked with internal standard. Corrective action for this is outlined in section 2.2.

2.1.3 If one or more of the surrogates are slightly out of control and none of the above situations appear to apply, the sample will be re-analyzed. If surrogates are still out of control, and there is no reason to assume the instrument is out of control, matrix interferences or preparation error will be assumed. Proceed as directed in 2.1.1.2.2.

2.2 Internal Standard Failure

2.2.1 Any time an internal standard fails the -50% +100% area criteria, the ability to accurately quantitate an analyte is lost. For that reason every effort will be made to prevent the failure of an internal standard. Internal standards can fail their area count criteria for the following reasons: software failure, matrix interferences, mis-spiked, or instrument failure.

2.2.1.1 Software Failure

2.2.1.1.1 Samples with poor matrixes or some sort of chromatographic interferences may cause the quantitation software to mis-integrate the peak. If an internal standard fails its area criteria under these circumstances, the data will be reviewed for mis-integration. If this solves the problem, no narration or qualification will be necessary, and the affected analytes will need to be requantitated; if not, proceed as directed below.

2.2.1.2 Matrix Interferences

2.2.1.2.1 If, even after manual integration, a matrix interference is preventing accurate internal standard area quantitation, the sample will be either:

a) re-analyzed, or most likely
b) diluted and re-analyzed

The correct course of action is dependent on the extent of the matrix interferences. Since specific compounds go with specific internal standards, if the sample is diluted, both the non-diluted and diluted runs will be combined and reported as one. The analysis date reported will be the date the initial run was made. The elevated detection limits reported for the compounds associated with the diluted run will be explained in a narration stating why the dilution was made. As long as the internal standard area is acceptable in the diluted run, no data qualifications will be made based on internal standard areas. Exceptions to the rule will exist when the non-

diluted run has matrix problems outlined elsewhere in this section. In these instances only the diluted run will be used.

2.2.1.3 Mis-Spiked

2.2.1.3.1 If surrogate recoveries and internal standard areas fail their respective criteria, the data will be reviewed for a possible mis-spike of the internal standard, if they were manually spiked. If upon review the analyst determines that the sample was mis-spiked with internal standard, the sample will be re-quantitated according to the concentration of the internal standard present in the sample. If the level of internal standard present in the sample cannot be determined, and the sample's hold time has not expired, the sample will be re-analyzed. No data will be qualified as long as the re-analysis works. If the sample is out of its' hold time, the project chemist will be consulted and the analyst will proceed as instructed, by either qualifying the data for that fraction as estimated based on poor internal standard recoveries, or by having the sample re-analyzed. If the re-analysis works, the data will be narrated and qualified because it was re-analyzed outside of the hold time. (See LIMS qualifier # 1).

2.2.1.4 Instrument Failure

2.2.1.4.1 If the internal standards and surrogate standards both fail their criteria, and the standards were added automatically by the instrument, the sample was probably mis-injected. Inspect the autosampler standard injection module to be sure that it is filled with the internal/surrogate standard mixture, as well as to verify that it is dispensing the mixture correctly. Once everything appears to be in functioning properly, then sample will be re-run. No narration or qualification will be necessary if the re-run works.

2.3 Dilution Procedures

2.3.1 Samples will be diluted when there is:

a) sufficient cause to believe that the ability to see the non-diluted detection limits is in question.

b) a chromatography problem from a matrix interference which may be interfering with the recovery of target analytes.

c) sufficient cause to believe that the matrix of the sample will be detrimental to the instrument.

2.3.2 Elevated detection limits will never be reported based on the belief that the non-diluted detection limits cannot be achieved. The sample will always be diluted and the elevated detection limits reported from the results of the diluted run.

2.3.3 The minimum dilution possible will be made that allows the analyte with the highest concentration to be kept near the upper half of the linear range of the instrument. If the dilution is being made based on matrix problems rather than elevated target analyte concentrations, the minimum dilution possible will be made. The sample will be re-run at a lesser dilution if initially over diluted to accomplish this.

2.3.4 Under normal operating conditions, if a sample was initially run straight and a dilution is required, all results and detection limits will be reported from the diluted run only (except as noted above in 2.2.1.2.1).

2.3.5 Diluted samples will be narrated explaining the reason for the elevated detection limits unless it is obvious due to the high target concentrations being reported. (See LIMS qualifier # 7).

2.4 Laboratory Fortified Blank (LFB) Failure

2.4.1 Failure of any part of an LFB requires qualification or when possible, re-analysis of the samples in that batch looking for the failed compound. If any compound or compounds in the Laboratory Fortified Blank (LFB) exceed the upper control limit for recovery, positive results for that analyte for every sample in the QC batch will be estimated. All results less than the detection limit are acceptable and need no qualification. Detection limits will not be qualified. (See LIMS qualifier # 66). However, if any compound or compounds in the LFB are out of control low, positive results for that analyte for every sample in the batch will be qualified as estimated and the detection limit for that analyte will be considered approximate. (See LIMS qualifier # 5). A batch narrative will be written any time a LFB analyte fails recovery criteria.

2.5 Matrix Spike (SPK1 and SPK2)/Matrix Spike Duplicate (MSD) Failure

2.5.1 If all analyte recoveries are within laboratory generated windows on both SPK1 and SPK2, but the precision for the MSD is out, the data for the failed compound in the non-spiked version of the sample will be qualified as estimated. (See LIMS qualifier # 3).

2.5.2 If the percent recovery for an analyte is in control in one SPK, out of control in the other SPK, and the MSD percent difference is in control, no data will be qualified, but a narrative will be written. (See LIMS qualifier # 63).

2.5.3 If any single analyte recovery is outside the laboratory generated windows on either or both SPK1 and SPK2, and the MSD precision is out, the data for the failed compound in the non-spiked version of the sample will be qualified as estimated. (See LIMS qualifier # 2).

2.5.4 If any spiked compound fails in the either or both SPK1 and SPK2, and in the LFB, the data for that analyte for every sample in the batch will be estimated. (See LIMS qualifier # 5).

2.5.5 If SPK1 and/or SPK2 percent recoveries for any analyte are above the upper control, and the results for that analyte in the sample are less than the reporting limit, then no qualification is required. (See LIMS qualifier # 68).

2.5.6 If a sample has a concentration of a spiked analyte that is greater than five times the spiked concentration, and the resulting spike recoveries for SPK1 and/or SPK2 and outside of the control limits, no qualification to sample results is necessary. (See LIMS qualifier # 86).

2.5.7 If the matrix spike recoveries are unable to be obtained due to high background matrix interferences, high analyte concentration, or interfering peaks, then the sample will be qualified for each analyte affected. (See LIMS qualifier # 23, 24, and 25 respectively.)

2.6 Method Preparation Blank (MPB) or Daily Instrument Blank (BLK) Failure

2.6.1 The MPB or BLK will fail for two reasons:

a) surrogate recoveries in the MPB or BLK are outside of the laboratory generated control limits
b) either the MPB or the BLK are contaminated with one or more target analytes

2.6.2 If the surrogate recoveries in the MPB or BLK are above the laboratory generated control limits, data will be narrated but not qualified. (See LIMS qualifier # 15). If surrogate recoveries are below the laboratory generated windows, the BLK must be viewed as mis-prepared and cannot be used to determine sample contamination from the purge and trap system. In this instance, the MPB or BLK must be re-prepared and re-analyzed.

2.6.3 If the BLK is contaminated with any target analytes above the reporting limits for that analyte, analysis may not begin. The problem must be corrected before sample analysis can re-commence.

2.6.4 Sample data will never be blank subtracted. Positive hits found in the BLK and the sample will be reported, and narrated in an analysis narrative. The data for that compound will be reported as estimated. (See LIMS qualifier # 21, 31, and 67.)

2.6.5 In most instances for volatiles, the BLK and the MPB will be reported using the same analysis. The exception to this is for highly concentrated methanol extracted samples. In this case the MPB is prepared and reported separately.

2.7 Missed Holding Times

2.7.1 All samples that are analyzed beyond their established USEPA maximum holding times must be qualified. All positive results must be qualified as estimated, and all non-detectable results must be qualified as approximate. (See LIMS qualifier # 1). If the hold time is missed due to laboratory error, a detailed explanation must be provided with the qualifier.

2.7.2 All samples that are received by the laboratory that have already exceeded their USEPA maximum allowable holding time must have their results qualified as estimated. (See LIMS qualifier # 12).

2.7.3 Trip blanks accompany the samples for many projects when volatile analysis is required. The purpose of the trip blank is to check for cross contamination from the samples in the project. For this reason, when a trip blank is analyzed outside of its USEPA maximum hold time, the sample will be narrated as having missed the holding time, but no qualifications are required to the results. (See LIMS qualifier # 59).

2.8 Improper Preservation of Samples

2.8.1 All volatile water samples are checked during analysis to see if they are properly preserved. Any samples found to have a pH of greater than 2 will be narrated as such, but no qualification of the results will be performed.

2.8.2 When analyzing for acrolein and acrylonitrile, samples will require special treatment.

2.8.2.1 When acrolein and acrylonitrile are requested for a 624 analysis, as specified in 40 CFR Part 136, Appendix A, the results should be considered as screening results only. All positive results will be qualified as estimated. (See LIMS qualifier # 29).

2.8.2.2 For analyses other than 624, samples collected for acrolein and acrylonitrile must be either preserved at a pH of 4-5 or analyzed within 3 days of collection. All results for these compounds are to be qualified or estimated if the above criteria are adhered to. (If only acrolein was requested LIMS qualifier # 30 is to be used. If both analytes are requested, then LIMS qualifier # 76 is used).

2.9 Storage Blanks

2.9.1 As a preventative check against cross contamination of samples by either samples in the same project, samples from another project, or background lab contaminants, a storage blank will be stored in all volatile refrigerators. The storage blanks will be removed after each week, and subsequently analyzed by GC/MS.

2.9.2 If any positive results are obtained for any analyte in the storage blanks, the Quality Assurance Office will be notified immediately.

Approved By: RL 11/5/97 QA Manager

Approved By: JD 11/5/97 Area Manager

2.10 TCLP Blanks

2.10.1 For each Zero Headspace Extraction (ZHE) unit used for TCLP extraction, a blank must be tumbled and analyzed once for every twenty extractions performed.

2.10.2 The ZHE tumble blank will then be analyzed by GC/MS to determine if the ZHE unit is free from contamination. If any contamination is found, the unit must be thoroughly cleaned, and shown to be free of contamination before being put back into service.

2.11 4-Bromofluorobenzene (BFB) Failure

2.11.1 BFB must be successfully analyzed following the instructions and criteria provided in the volatile mass spectrometry SOP (GR-04-104) prior to the analysis of any samples. If BFB does not pass the required criteria, the following steps should be considered, in order, in an effort to solve the problem. Under no circumstances will analyses be performed until BFB passes the required mass abundance criteria.

2.11.1.1 Re-analyze the BFB run. This frequently works, especially when the instrument has set idle over the weekend.

2.11.1.2 Depending on the masses that have failed the abundance criteria, try a second re-analysis or re-tune the instrument.

2.11.1.3 Re-tune the instrument again.

2.11.1.4 Depending on the outcome of each analysis following the re-tune, the analyst may continue re-tuning and re-analyzing until a successful BFB is analyzed. Analyst experience will determine when the instrument is behaving such that re-tuning is the appropriate course of action vs. cleaning the source.

2.11.1.5 Shut down the instrument and clean the source.

3.0 REPORTING AND DELIVERABLES

3.1 All out of control data must be narrated or qualified as appropriate, using the conventions required by the laboratories LIMS system. It is extremely important that the end user of the data be informed of all data which requires some sort of qualification or narration.

4.0 QUALITY ASSURANCE

4.1 Quality assurance procedures are covered in section 2.0.

Approved By: _____ 11/5/97
QA Manager

Approved By: _____ 11/5/97
Area Manager

5.0 REFERENCES

5.1 For further information, consult appropriate volatile laboratory method SOP, or the TriMatrix Laboratories, Inc. Quality Assurance Manual.

5.2 USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review. Office of Emergency and Remedial Response, U.S. Environmental Protection Agency, Washington, DC 20460. Publication 9240.1-05, PB94-963501, EPA540/R-94/012, February, 1994.

6.0 ATTACHMENTS/APPENDICES

6.1 Not applicable.

Approved By: _____ 11/5/97 **Approved By:** _____ 11/5/97

QA Manager **Area Manager**

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

EXTRACTION OF SEMI-VOLATILE BNA'S IN SOIL, SEDIMENT AND SLUDGE

USEPA METHOD 3550 Modified

APPROVALS:

Prep Lab Supervisor: Randy Scott Date: 1/18/96

QA/QC Supervisor: Scott Date: 1-23-96

Laboratory Manager: Date: 1/18/96

Procedure Number: GR-09-103

Revision Number: 1.0 By: Randy Scott Effective Date: 1/18/96

Total Number of Pages: 9 Pages Revised: All

TriMatrix Laboratories, Inc.
Standard Operating Procedure

Subject:	Extraction of Semi-Volatile BNA's in Soil, Sediment and Sludge	Procedure No: GR-09-103 Revision No:1.0 Effective Date: 1/18/96
	USEPA Method 3550 Modified	*Page 1 of 9*

1.0 METHOD SUMMARY

1.1 Low/Medium Concentration Method

A 30 g sample is mixed with anhydrous sodium sulfate to form a free flowing powder. The sample is serially sonicated with 1:1 methylene chloride/acetone, and concentrated to a 1 ml volume. The extract is analyzed by GC/MS. This method is for the extraction of low to medium concentration of analytes. If past data or poor sample matrix shows the sample(s) to be inappropriate for this method then final extract volumes may be greater than 1 ml or the high concentration method will be used (see 1.2).

1.2 High Concentration/Poor Sample Matrix Method

This method is intended for samples that have a history of high analyte concentrations and/or poor sample matrices that typically contain high concentrations of hydrocarbons. The amount of sample that is sonicated varies between 2 and 10 g depending on the individual sample. The extraction analyst will use his/her professional experience (and past history data for the sample if available) to determine the initial amount to extract. The extraction procedure will be the same as the low/medium method with the exception of initial weights, using only methylene chloride as the solvent and possibly final volumes. The final volume will depend on the sample matrix.

2.0 SAFETY PRECAUTIONS

2.1 For extractions and surrogate/spike standard preparation, follow the safety procedures as outlined in the latest edition of the Laboratory Safety Manual.

2.2 For proper sample and spike waste storage and disposal, or spill response, see the latest edition of the Laboratory Waste Disposal SOP.

3.0 APPARATUS

3.1 600 ml heavy duty Pyrex beaker.

Approved By: SJ 1-23-96 **QA/QC Supervisor** **Approved By:** [signature] 1/18/96 **Area Supervisor**

3.2 Sonicator:
1. Tekmar Sonic Disrupter Model number TM500 or TM600, with 3/4 inch horn.
2. Fisher Model No. 550 with 3/4 inch horn.

3.3 500 ml Kuderna-Danish flask.

3.4 10 ml graduated concentrator tube.

3.5 3-ball macro Snyder column.

3.6 Variable temperature steam bath.

3.7 Top loading balance, capable of accurately weighing to the nearest .01 g.

3.8 N-EVAP or similar nitrogen blowdown device.

3.9 Whatman # 4, 24 mm filter paper (or equivalent).

3.10 Stainless steel spatula.

3.11 1 ml disposable glass Pasteur pipets.

3.12 Boiling chips - solvent rinsed, approximately 10 mesh (silicon carbide or equivalent) (Hengar micro granules).

3.13 2 ml auto-sampler vials calibrated to 1 ml, with Teflon lined silicon septa, and screw cap lids.

3.14 1 ml syringe.

3.15 Grinder, or alternative size reduction tools.

3.16 Mortar & pistal

4.0 CHEMICALS AND REAGENTS

4.1 1:1 mix of Methylene Chloride/Acetone - pesticide quality or equivalent.

4.2 Methylene chloride - pesticide grade or equivalent.

4.3 Anhydrous Sodium sulfate (ACS granular). Baked @ 400°C for 4 hours.

4.4 See Appendix A for surrogate and/or spiking standard preparation.

5.0 PROCEDURE

5.1 Sonication

5.1.1 Tuning the Sonicator(s)

5.1.1.1 The sonicator(s) must be tuned prior to use at the beginning of the analysts shift.

5.1.1.2 Follow the tuning instructions starting at sec. 5.1.1.2.1 for the Tekmar TM500 or TM600. For the Fisher model 550 follow the instructions starting at sec. 5.1.1.2.9.

5.1.1.2.1 **Tuning (Tekmar TM500 and TM600)**
Tune the power supply in accordance with the following procedure each time the sonicator is used.

5.1.1.2.2 Ensure that the horn is not immersed in liquid and that it does not come in contact with anything.

5.1.2.2.3 If using two converters simultaneously, disconnect one

converter from the power supply.

5.1.2.2.4 Set the tuning select switch to the down position if the remaining converter is connected to connector ; and to the up position, if the remaining converter is connected to connector .

5.1.2.2.5 Set TIMER to HOLD.

5.1.2.2.6 Set OUTPUT CONTROL to 10 (to 5 when using a microtip).
CAUTION: When using a micro tip, never allow the tip to vibrate in air for more than 10 seconds and do not permit the OUTPUT CONTROL setting to exceed number 5. Ignoring these instructions could cause the microtip to fracture.

5.1.2.2.7 Momentarily hold down ON/OFF/TUNE switch to TUNE and rotate the tuning control clockwise or counterclockwise until a minimum (not maximum) reading is obtained on the loading meter . If the tuning control strikes the limit stops before the meter reads minimum and starts to rise again, the power supply is not tuned. If minimum reading (sometimes referred to as a null) cannot be obtained, the horn, coupler section, tip, micro tip, or lower section is out of resonance or the power supply and converter require servicing.
NOTE: If minimum reading cannot be obtained and another horn is available, check unit with new horn to ascertain whether the power supply or horn is at fault.

5.1.2.2.8 Release ON/OFF/TUNE switch. The sonicator is now tuned. Record the tuning information in the TUNE logbook.

5.1.1.2.9 **Tuning Instructions for the Fisher Model 550 Sonicator**

5.1.1.2.10 Turn OUTPUT CONTROL knob counterclockwise to zero.

5.1.1.2.11 Press POWER SWITCH to ON (up) position. The switch will illuminate.

5.1.1.2.12 When the prompt appears, press TUNE key. Screen will read: [TUNING - - - PROBE ACTIVE].

5.1.1.2.13 Turn the OUTPUT CONTROL knob towards setting 10 (5 if using a microtip).
(a) Note the position of the Bar Graph on the ICD Display Screen. Do NOT exceed 70%.
(b) Rotate the Tuning Knob clockwise or counterclockwise until a minimum (not maximum) reading is obtained. Note: If a reading of less than 15% can not be obtained there is a problem with teh sonicator and it must be fixed before it can be used.

5.1.1.2.14 Press the TUNE key to display prompt for programmed or continuous operation and set the sonicator for programmed operation.

5.1.1.2.15 The sonicator is not tuned. Record all tuning information in TUNE logbook.

5.1.1.2 The sonicator(s) must be tuned prior to use at the beginning of the analyst shift.

5.1.2 Decant and discard any water layer and any foreign objects such as sticks, leaves, and rocks from the soil sample. Mix sample thoroughly, especially composite samples. The sample should be able to pass through a 1 mm sieve. If it appears that the sample will not, then it must be size reduced using a mortar and pistal or

mechanical grinder.

5.1.3 If the sample is gummy, fibrous, or an oily type sample then it should be size reduced to allow for the maximum surface area exposure to the solvent. Material such as paper or fabric need not be size reduced. The analyst needs to use their professional judgment to determine the proper handling of the sample.

5.1.4 Label all glassware with complete sample number and other necessary information (i.e. MS, MSD, LFB, etc.).

5.1.5 The following steps should be performed rapidly to avoid loss of the more volatile compounds. For sandy to dry samples, weigh 30 ±0.1g of sample into a 600 ml beaker and add ~20-25 g of anhydrous sodium sulfate. For samples that are of a wet to gummy nature, add ~60 g of anhydrous sodium sulfate to the 30 ± 0.1 g of sample. Mix well. The sample should have a sandy texture at this point. Add more anhydrous sodium sulfate as necessary to achieve this consistency. Add 1.0 ml BNA surrogate to the samples, blanks and spikes. For matrix spikes and laboratory fortified blanks, also add 1.0 ml of spiking standard. Immediately add 100 ml of the 1:1 methylene chloride/acetone mix.

5.1.6 Place the 3/4 inch tipped sonication probe about 1/2 inch below the surface of the solvent but above the soil layer.

5.1.7 Sonicate for 3 minutes at full power (output control knob set at 10 and percent-duty cycle knob set at 50%). Do not use a micro tip probe.

5.1.8 Assemble the K-D apparatus. Insert a 100 mm filtering funnel lined with 24 mm # 4 filter paper. Add ~3/4 to 1 inch of the anhydrous sodium sulfate into the funnel. Rinse filter paper and sodium sulfate with methylene chloride.

5.1.9 Decant the solvent into the filtering funnel.

5.1.10 Repeat the sonication two more times with two additional 100 ml portions of 1:1

methylene chloride-acetone, decanting off the solvent after each sonication. If, after the third extraction, there is no noticeable color change in the extract, additional sonications may be needed. Record the exact number of sonications in the soil log book, if additional sonications were needed. On the final ultrasonic extraction, pour the entire sample into the filtering funnel and rinse with methylene chloride.

5.2 Concentration

5.2.1 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding ~1 ml methylene chloride to the top. Place the K-D apparatus on a hot water bath (80-90°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 ml (usually about 10 to 15 minutes), remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.

5.2.2 Place the concentrator tube in the N-EVAP (water bath ~35°C) and evaporate the solvent volume to 1 ml using a gentle stream of clean, dry nitrogen.

CAUTION: When the volume of solvent is reduced below 1 ml, semivolatile analytes may be lost. If the extract is allowed to concentrate to, or near dryness, the entire extraction must be repeated.

5.3 Transfer the extract into a 1.7 ml auto-sampler vial which has been calibrated to 1 ml, (see SOP for vial calibration) making sure to rinse the concentrator tube and side walls 2-3 times with a total of ~ 500 µl methylene chloride.

5.4 Place the auto-sampler vial in the N-EVAP (water bath ~35°C) and evaporate the solvent volume to 1 ml using a gentle stream of clean, dry nitrogen.

5.5 Cap the vial with the screw cap lids and Teflon lined silicon septa,and place the 1 ml extract in the GC/MS refrigerator at 4°C.

6.0 FLOW CHART

Not Available.

7.0 QUALITY ASSURANCE

7.1 A method blank must be extracted at the start of each working shift. The blank is defined by weighing 30g of sodium sulfate and extracting it using the same procedure that will be used for the samples. The method blank that is extracted at the start of every shift may be used to start a QC batch including a LFB, MS, MSD or may be placed within an open QC batch with less than 20 samples in it.

7.2 A quality control set consisting of a method blank, matrix spike, matrix spike duplicate (provided enough sample was received), and laboratory fortified blank will be extracted at a minimum of every 20 samples per matrix to evaluate laboratory data quality. Performance records are maintained to document the quality of the data that is generated.

7.3 Matrix spikes are prepared by adding 1 ml of the spike standard in addition to the 1 ml of BNA surrogate standard to the sample, and extracting the sample as outlined beginning in Section 5.1.

7.4 Laboratory fortified blanks are prepared by adding 1 ml of the spike standard in addition to the 1 ml of BNA surrogate standard to 30 g of anhydrous sodium sulfate, and extracting the sample as outlined beginning in Section 5.1.

7.5 Interferences

7.5.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware. Rinsing all washed glassware with pesticide quality methylene chloride prior to use, as well as the use of high

purity reagents and solvents helps to minimize interference problems.

7.6 Matrix interferences from either the sample itself, or from laboratory induced contamination may effect the recovery of both analytes and spiked compounds. Re-extraction of a sample or spike may be necessary if any of the surrogate or spike compounds fail to pass the appropriate recovery limits.

8.0 RESPONSIBILITIES

The extraction chemist is responsible for all standard preparation, sample set-up and extraction, surrogate spiking and standard additions, and sample drying, concentration, and storage. The extraction chemist must also maintain a detailed laboratory notebook and fill out all appropriate benchsheets. The extraction chemist is also responsible for proper waste disposal of all spent solvents from extractions, and appropriate spill response procedures.

9.0 REFERENCES

Test methods for ultrasonic extraction: SW-846 Method 3550A, September 1994, Rev. 1.

10.0 APPENDIXES

Appendix A: Surrogate/Spiking standard preparation for GC/MS and GC laboratories.

Approved By: SJ 1-23-96 QA/QC Supervisor

Approved By: Randy Scott 1-18-96 Area Supervisor

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

SEMI-VOLATILE LABORATORY
ION TRAP MASS SPECTROMETRY ANALYSES OF
BASE/NEUTRAL/ACID COMPOUNDS

MODIFIED METHODS 8270C AND 625

APPROVALS:

Organics Laboratory Manager: _____ Date: 12/14/98
Jeff P. Glaser

QA Manager: _____ Date: 12/14/98
Rick D. Wilburn

Laboratory Manager: _____ Date: 12/14/98
Douglas E. Kriscunas

Procedure Number: GR-04-103
Revision Number: 4.0

Date Initiated: 12/9/98
Effective Date: 12/9/98

Date Revised: New
Pages Revised: New

By: Janet M. Kudirka

Total Number of Pages: 45

1.0 SCOPE AND APPLICATION

1.1 Method 8270C is used to determine the concentration of semi-volatile organic compounds in extracts prepared from all types of solid waste matrices, soils, and ground water. Direct injection of a sample may be used in limited applications.

1.2 Method 8270C is used to quantify most basic, neutral, and acidic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic using a fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols. See Table 1 for target compounds, retention times, quantitation and secondary ions, internal standards and surrogates typically included in a Method 8270C analysis. Table 1A provides additional Appendix IX and other analytes covered by this method.

1.3 The following compounds may require special treatment when being determined by this method. Benzidine can be subject to oxidative losses during solvent concentration. Its chromatography must be monitored for peak broadening. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition. N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. 1,2-diphenylhydrazine also decomposes in the injection port into azobenzene. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with a high boiling material.

1.4 The default quantitation limits for clean samples extracted from both 1000 mL or 30 g of sample, concentrated to 10 ml, and analyzed by Method 8270C are given in Table 2 and 2A. Quantitation limits will be proportionately higher depending on sample matrix and preparation method, and for sample extracts that require dilution.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method (see section 19).

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update III, Method 8270C, "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 3, December 1996.

2.2 *40 Code of Federal Regulations, most current edition*, Pt. 136, App. A, Method 625-Base/Neutrals and Acids.

3.0 SUMMARY OF PROCEDURE

3.1 Prior to using this method, the samples should be prepared for analysis using the appropriate sample preparation and cleanup methods. Waters will be extracted using Modified EPA method 3510C, and soils by Modified EPA Method 3550B. This SOP describes the analysis of the resulting extract.

3.2 This SOP will be used as the default Method for all mass spectrometry ion trap semi-volatile analyses. It has been written to follow Method 8270C as closely as possible, and has been customized to cover the equipment and techniques being used at TriMatrix Laboratories, Inc., Grand Rapids, Michigan.

3.3 Wastewater samples will use the following Method 625 modifications.

3.3.1 The 12 hour shift is replaced by a 24 hour shift. DFTPP and a continuing calibration standard are analyzed once every 24 hours instead of every 12.

3.3.2 A 3 point curve can be used in place of a 5 point curve. SPCCs and CCCs are not used. Instead the RFs for every compound of interest listed on the Method 625 list must have <35% RSD for the curve to be valid.

3.3.3 The continuing calibration standard also does not use SPCCs and CCCs. The RF for every compound of interest in the continuing calibration standard that is on the 625 list must be ≤ 20% difference when compared against the curve. If not, either a new continuing calibration standard will be run with acceptable percent differences, or a new 3 or 5 point curve must be run.

3.3.4 There are no criteria for internal standard areas or retention times in Method 625. The Method 8270C criteria will be followed.

3.3.5 Every compound of interest that is on the Method 625 list (Method 625 Table 6) must be in the matrix spikes and the laboratory fortified blanks, not just the limited list specified in Method 8270C. Laboratory generated windows for spike recoveries are calculated as specified in the 8270C SOP. Additionally requested compounds are not spiked.

4.0 PARAMETER OR COMPOUND LIST

4.1 Analytes available for analysis by Method 8270C are listed in Table 1 and 1A. Other analytes may be run by this method provided they have been developed following the guidelines in this SOP

5.0 REFERENCED SOPs

5.1 TriMatrix Laboratories SOP #GR-09-101, *The Extraction of BNAs in Water*, latest version.

5.2 TriMatrix Laboratories SOP #GR-09-103, *Extraction of Semi-Volatile BNAs in Soil, Sediment and Sludge*, latest version.

5.3 TriMatrix Laboratories SOP #GR-04-101, *Semivolatile Laboratory Mass Spectrometry Corrective Actions*, latest version.

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the total ion current profiles. All materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. Rinsing all washed glassware with pesticide quality or better methylene chloride immediately prior to use, as well as the use of high purity reagents and solvents helps to minimize interference problems.

6.2 Contamination can be caused whenever highly concentrated samples are followed by low or non-detect samples. This phenomenon is called carryover. To reduce carryover, the auto sampler syringe is washed multiple times between samples. Whenever possible, the analyst should analyze a reagent blank immediately after a high level sample. If carryover is suspected, all affected samples should be re-analyzed.

6.3 Corrective actions are outlined in TriMatrix Laboratories SOP #GR-04-101, *Semivolatile Laboratory Mass Spectrometry Corrective Actions*, latest version.

7.0 SAFETY PRECAUTIONS

7.1 The analyst must comply with all standard operating procedures for health and safety as outlined in the TriMatrix Laboratories, Inc. Laboratory Safety Manual.

7.2 Methylene chloride and most of the target analytes, surrogates, and spikes, are highly toxic and cancer suspects. Wear appropriate protective clothing and gloves to avoid contact. Prepare standards and samples under a fume hood.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 To achieve the quantitation limits described in this method a minimum of 30 g of soil and 1 liter of liquid sample must be collected. More sample will be necessary if sample matrix spikes will be required.

8.2 Samples should be collected in cleaned glass containers with screw cap lids and Teflon liners. Plastic containers and/or plastic lined lids may NOT be used due to potential phthalate contamination.

8.3 Samples are stored at 4°C, and sample extracts are stored between -10 and -20°C and protected from light.

8.4 Analysis must be performed within 40 days of sample extraction.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Varian Gas chromatograph/mass spectrometer system

9.1.1 Varian Saturn II ion trap mass spectrometer is set up to scan from 40-450 mass units twice per second, using 70 volts (nominal) electron energy in the electron impact mode, and producing a mass spectrum that meets all the criteria in Table 3 when 5 ng of decafluorotriphenylphosphine (DFTPP) are injected onto the analytical column.

9.1.1.1 MS operating conditions:

- Electron energy: 70 volts (nominal).
- Mass range: 40-450 amu.
- Scan time: 820 amu/second = 2 scan/sec.
- Source temperature: 190°C
- Transfer line temperature: 250°C

9.1.1.2 GC operating conditions:

- Column Temperature Program: 45°C for 1.5 min, then to 200°C at 10°C/min, then to 315°C at 25°C/min, hold at 315°C until benzo[g,h,i]perylene has eluted
- SPI Injector Temperature Program: 45°C for 0.5 min, then to 300°C at 100°C/min
- Sample volume: 1 ul
- Carrier Gas: ultra high purity helium at 8psi (~2-3 ml/min)

9.1.2 Injector

9.1.2.1 A SPI (Septum Programmable Injector) temperature programmed injector that allows sub-ambient initial injector temperatures followed by rapid heating up to 315°C

9.1.3 Fused Silica Capillary Column: 30m x 0.32mm i.d. 1 um film thickness silicon coated DB-5MS or equivalent.

9.1.4 Data system: PC based multi-tasking computer that allows the continuous acquisition and storage on magnetic tape or compact disk of all mass spectra obtained throughout the duration of the chromatographic program. The software can search any GC/MS data file that was

acquired on the system for ions of a specific mass, and can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software allows integrating the abundances in any EICP between specified time or scan number limits. The 1992 version of the NIST mass spectral library is available for the identification of unknowns.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 When chromatography degrades or sensitivity decreases, routine maintenance consists of cleaning/changing injection port liner, clipping the capillary column, changing the injection port septa, and cleaning the ion trap.

11.0 CHEMICALS AND REAGENTS

11.1 All chemicals and reagents used must be pesticide grade or better, and must be determined to be free of interferences.

11.2 Acetone, Hexane, Methylene Chloride, Toluene - pesticide grade or equivalent.

12.0 STANDARDS PREPARATION

12.1 Stock quantitation standard solutions: Standard solutions are prepared from pure standard materials or purchased as certified solutions.

12.1.1 Commercially prepared stock standards are used to prepare the majority of quantitation working standards. They commonly come as mixes of compounds at 2000 mg/L sealed in various glass ampules that are combined during preparation. This mix is combined with the surrogate standard that is currently being used during sample extraction to create an 8270C quantitation standard. This standard is made up at a concentration of 200 mg/L using methylene chloride as the solvent. Dilutions of this 200 mg/L standard make up the 6 point initial calibration curve at concentrations of 25, 20, 15, 10, 5 and 2 mg/L. Benzoic acid and Benzidine responses are not linear in this concentration range, so extra stock standard of each of these is added. The range for Benzoic acid is 10-125 mg/L and 6-75 mg/L for Benzidine.

12.1.2 Lab made stock standard solutions from neat compounds are prepared by accurately weighing out the analyte to four significant figures. The material is dissolved in methylene chloride or other suitable solvent and diluted to volume in a volumetric flask. When compound purity is assayed to be 96% or greater, the weight is used without correction to calculate the concentration of the stock standard.

12.1.3 The stock standard solutions are transferred into screw-cap bottles with Teflon lined lids, stored at -10 to -20°C and protected from light. Stock standard solutions are monitored for signs of degradation or evaporation.

12.1.4 All stock standard mixes will be replaced annually or sooner if comparison with quality control check standards indicate a problem. Initial calibration curves and all dilutions of stocks except for the continuing calibration standards will be replaced every six months or sooner if comparison with quality control check standards indicate a problem. Continuing calibration standards will be re-diluted from stock weekly.

12.2 Internal standard solutions: The six internal standards used are 1,4-dichlorobenzene-d_4, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, naphthalene-d_8, and perylene-d_{12}. The stock standard is prepared at 1000 ng/uL by dissolving 100 mg of each compound with a small volume of methylene chloride and then diluting to 100 mL as described in 12.1.2. Each 1 mL sample extract undergoing analysis is spiked with 5 uL of the internal standard solution, resulting in a concentration of 5 ng/uL of each internal standard.

12.3 GC/MS tuning standard: The standard used to tune the mass spectrometer contains 5 ng/uL of decafluorotriphenylphosphine (DFTPP), and 50 ng/uL of pentachlorophenol, benzidine, and 4, 4' DDT, in methylene chloride. It is prepared as described in 12.1.2, and is used to tune the instrument, to verify injection port inertness, and to monitor GC column performance.

12.4 Surrogate standards: The surrogate standards used are o-terphenyl, 2-fluorophenol, phenol-d_6, 2,4,6-tribromophenol, nitrobenzene-d_5, and 2-fluorobiphenyl. These are prepared in methanol in the same manner as described in section 12.1.2. The base/neutral surrogate spike is made up at 200 mg/L and the acid surrogate spike is made up at 400 mg/L in methanol according to Method 3500. It is then combined for a base/neutral concentration of 100 mg/L and an acid concentration of 200 mg/L. The amount spiked is 100 uL per each one mL of final extract volume, resulting in a concentration of 10 ng/uL of base/neutral surrogates and 20 ng/uL acids per each mL of extract.

12.5 Matrix spike standards: The compounds in the base/neutral matrix spike solution are acenaphthene, 1,4-dichlorobenzene, 2,4,-dinitrotoluene, naphthalene, N-nitrosodi-n-propylamine, pyrene, and 1,2,4-trichlorobenzene. The acid spike contains 4-chloro-3-methyl phenol, 2-chlorophenol, 4-nitrophenol, pentachlorophenol, and phenol. Refer to section 12.1.2 for preparation techniques. The spikes are made up in methanol at concentrations of 100 mg/L for the base/neutral compounds and 200 mg/L for the acid compounds according to Method 3500. The standards are spiked in at 100 uL per each 1 mL of final extract volume resulting in a concentration of 10 ng/uL for the base/neutrals, and 20 ng/uL for the acids.

Note: For all State of Wisconsin samples, every analyte of interest must be spiked when preparing and analyzing matrix spikes and matrix spike duplicates. Spike amounts and concentrations will be the same as that specified above.

13.0 SAMPLE PREPARATION

13.1 For extraction procedures, see the appropriate Extraction Lab SOP, referenced in section 5.0.

13.2 All samples to be analyzed must be spiked with surrogates. See section 12.4 for specifics.

13.3 All samples are spiked with internal standard immediately prior to analysis. See section 12.4 for specifics.

13.4 Unknown or potentially highly contaminated extracts can be screened using a GC/FID with a 30m x 0.32mm i.d. DB-5 or equivalent capillary column with a 1 um film thickness. This minimizes contamination of the GC/MS system from unexpectedly high concentrations of organic compounds and/or matrix interferences.

14.0 CALIBRATION PROCEDURES

14.1 Set up instrument as specified in section 9.1.1.1 and 9.1.1.2.

14.2 All samples and standards are introduced into the system via direct injections. Manual injections are performed with a 10 uL syringe. Automated injections are performed using a Leap autosampler. 1 uL injections are performed on all samples and standards.

14.3 Each GC/MS system is hardware-tuned to meet the criteria in Table 3 for a 5 ng, 1 uL injection of DFTPP. No analyses may begin until these criteria are met. Spectra are taken across the apex of the 198 ion. Background subtraction is designed to only eliminate column bleed or instrument background ions. The GC/MS tuning standard is also used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%. Calculate the % breakdown as follows:

$$\%\ \text{degradation} = \frac{\text{Area}_{DDE} + \text{Area}_{DDD}}{\text{Area}_{DDE} + \text{Area}_{DDD} + \text{Area}_{DDT}} \times 100$$

14.3.1 This result will be recorded on the daily instrument logbook.

14.3.2 Pentachlorophenol and benzidine should be present at their normal response, with minimal peak tailing. If peak tailing appears suspect, a tailing factor will be calculated on the total ion chromatogram of pentachlorophenol, following the instructions provided on Figure 1, to determine if corrective action is required. A tailing factor of 1 to 2 is considered acceptable, a tailing factor of 2 to 4 is considered to be the warning limit, and a tailing factor of 4 to 5 is considered unacceptable requiring corrective action. If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to clip off the first 6-12 inches of the capillary column.

14.4 All analytes will be quantitated using the internal standard technique. Analytes and their corresponding internal standards are provided in Table 1 and 1A. For analytes not listed in either Table, the internal standard used for quantitation should be the one that elutes nearest the analyte. The ions used for quantitation are also provided in Table 1 and 1A. Only if interferences are noted can a secondary ion be used for quantitation. In this instance the RF for that compound must also be recalculated before any quantitation can take place.

14.5 Initial Calibration:

14.5.1 Calibration standards: A minimum of 5 points must be in the initial calibration curve. Table 1 calibration standards are typically run at levels of 2, 5, 10, 15, 20 and 25 ng/ul. The high, 25 ng/ul standard may be omitted; however, this is done at the expense of the linear range of the instrument. Each 1 mL aliquot of calibration standard is spiked with 5 uL of the internal standard solution prior to analysis. Other analytes, including those listed in Table 1A are run at appropriate concentrations based on the response of the analyte. The low point of the initial calibration curve must be at or below the required quantitation limit.

14.5.2 1 uL of each calibration standard (that also contains surrogates and internal standards) is analyzed. Figure 2 shows a chromatogram, with the proper chromatography, of a calibration standard containing the Method 8270C base/neutral and acid analytes. A Response Factor (RF) is calculated for each compound using the quantitation ion given in Table 1 or 1A, and the following formula:

$$RF = \frac{A_X \times C_{IS}}{A_{IS} \times C_X}$$

where:

A_X = Area of the characteristic ion for the compound being measured
A_{IS} = Area of the characteristic ion for the specific internal standard
C_X = Concentration of the compound being measured (ng/ul)
C_{IS} = Concentration of the specific internal standard (ng/ul)

14.5.3 A system performance check must be performed to ensure that average RFs are met before the calibration curve can be used. The System Performance Check Compounds (SPCCs) are listed in Table 4. The minimum acceptable average RF for the SPCC compounds is 0.050. The SPCC compounds typically have very low RFs (0.1-0.2) and tend to decrease in response as the chromatographic system or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated. The minimum RF for all non-SPCC compounds that are analytes of interest is ≥ 0.01.

14.5.4 The percent relative standard deviation is calculated for each compound using the following formula:

$$\%RSD = \frac{SD}{RF} \times 100$$

where:

SD = The standard deviation of the response factor for that analyte across the initial calibration curve.

RF = The average of the response factor for that analyte across the five point curve.

14.5.5 The percent RSD for each compound should be less than 15%. However, the percent RSD for each individual calibration check compound (CCC) MUST be less than 30%. If the percent RSD is greater than 30% for any CCC, corrective action, including replacing the injection port liner, replacing or clipping the capillary column, and recalibration of the GC/MS system, must be initiated.

14.5.6 The relative retention time of each compound of interest in each calibration run should agree within 0.06 relative retention time units.

14.5.7 If the percent RSD of any non-CCC compound is 15% or less, the relative response factor is assumed to be constant over the calibration range, and the average response factor should be used for quantitation.

14.5.8 If the percent RSD for any compound is greater than 15%, calibration curves should be constructed for those compounds by plotting the area ratio ($Area_{analyte}/Area_{IS}$) versus the concentration. A first order or higher regression fit should be used for the five calibration points. The analyst should select the regression order that gives the least amount of calibration error.

NOTE: It is not the intent of the method to allow non-linear calibration to be used to compensate for proper instrument maintenance. Thus, non-linear calibration must not be employed for methods or instruments previously shown to exhibit linear calibration for an analyte.

14.6 Continuing Calibration:

14.6.1 Prior to analysis of samples or standards, the DFTPP tuning standard is analyzed and evaluated as specified in section 14.3. These criteria must be demonstrated at the beginning of each 12-hr shift.

14.6.2 Immediately following every acceptable DFTPP analysis, continuing calibration standard(s) are run at a mid-level concentration. For the 8270C analytes listed in Table 1, the continuing calibration standard is run at 10 ng/ul. This standard contains all semi-volatile target analytes, including all required surrogates and internal standards. Other analytes, including those provided in Table 1A, are run at a continuing calibration standard concentration appropriate for the response of the compound. The response factors from the standards are compared with the average response factor from the initial calibration run on the same instrument.

14.6.3 System Performance Check Compounds (SPCCs): A system performance check is made on every continuing calibration standard. The SPCC criteria must be met in order for the continuing calibration standard to pass. The SPCC compounds listed in Table 4 are used for the continuing calibration run. The minimum RF for semi-volatile SPCCs is 0.050. The minimum RF for all non-SPCC compounds of interest is 0.01. If the minimum response

factors are not met, the system is evaluated, and corrective action is taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, or active sites in the column or chromatographic system. This check must always be met before analysis begins.

14.6.4 Calibration Check Compounds (CCCs): After the system performance check is met, the CCCs listed in Table 5 are used to check the validity of the initial calibration. Calculate the percent difference when using the average response factor model calibration:

$$\% \text{ Difference} = \frac{RF_v - RF_m}{RF_m} \times 100$$

where:

RF_v = response factor from verification standard
RF_m = average calibration factor from initial calibration

Use percent drift when calibrating using a regression fit model:

$$\% \text{ Drift} = \frac{C_1 - C_C}{C_1} \times 100$$

where:

C_1 = CCC Standard Concentration
C_c = measured concentration using appropriate quantitation method.

14.6.5 If the percent difference or drift for each CCC is less than ≤20%, the initial calibration is assumed to be valid. If any one CCC has a >20% difference from the initial calibration curve, corrective action must be taken. Problems similar to those listed under section 14.6.3 could also affect the CCC criteria. If the source of the problem cannot be determined after corrective action has taken place, the instrument is considered out of control. At a minimum the continuing calibration standard must be re-run. If necessary, a new curve will be run. The CCC criteria must also always be met before sample analysis begins.

NOTE: It is not the intent of the method to allow non-linear calibration to be used to compensate for proper instrument maintenance. Thus, non-linear calibration verification must not be employed for methods or instruments previously shown to exhibit linear initial calibration for an analyte.

14.6.6 The internal standard responses and retention times in the continuing calibration verification standard(s) and samples must be evaluated for each run. If the retention time for any internal standard varies by more than 30 seconds from the last calibration verification standard, the system must be inspected for malfunctions, and corrective action performed as necessary. If the EICP area for any of the internal standards varies by a factor of 2 (-50% to +100%) from

the midpoint standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrective action performed as necessary. All samples analyzed under either of these out of control conditions musts be re-analyzed.

15.0 ANALYTICAL PROCEDURE

15.1 Every 12 hour shift **must** begin with the analysis of DFTPP, followed by either a 5 point curve or a continuing calibration standard, and then a blank.

15.2 DFTPP **must** pass the required tuning criteria specified in section 14.3 before any subsequent analysis can begin. There is a 12 hour window that all acquisitions **must** be started in, beginning at the injection time of DFTPP. The entire 12 hour shift must be run under the same mass spectrometer conditions used for DFTPP. All samples must be run under the same gas chromatographic conditions as the quantitation standard. See 9.1.1.1 and 9.1.1.2 for recommended operating conditions.

15.3 Any compound requiring quantitation under Method 8270C **must** have an average Response Factor (RF) generated from a 5 point curve, and that curve **must** pass the required System Performance Check Compounds (SPCCs), and the Calibration Check Compounds (CCCs) specified in section 14.5.

15.4 A continuing calibration standard is run on days that a curve is not, and it also **must** pass the required criteria listed in section 14.6.

15.5 The analytical system **must** be demonstrated to be free from contamination under the conditions of the analysis by running a blank at least at the beginning of every 12 hour shift (see section 18.3).

15.6 A matrix spike, matrix spike duplicate, and laboratory fortified blank **must** be run for each quality control batch (up to a maximum of 20 samples/batch) to assess accuracy. Additional quality control method requirements are specified in the Quality Assurance section, 18.0.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Qualitative analysis:

16.1.1 An analyte is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). These standard reference spectra are obtained through analysis of the calibration standards. Two criteria must be satisfied to verify identification: (a) elution of sample component at the same GC relative retention time (RRT) as the standard component; and (b) correspondence of the sample component and the standard component mass spectrum.

16.1.1.1 The sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component in the daily continuing calibration standard. If co-elution of interfering components prohibits accurate assignment of the sample

component RRT from the total ion chromatogram, the RRT is assigned by using extracted ion current profiles for ions unique to the component of interest.

16.1.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%), are automatically checked by the software to be present in the sample spectrum. The relative intensities of ions must agree within plus or minus 30% between the standard and sample spectra. For example, an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.

16.1.2 For samples containing components not associated with the calibration standards, a library search can be made for the purpose of tentative identification. Only after visual comparison of sample spectra with the nearest library searches will the analyst assign a tentative identification. Guidelines for making tentative identification are:

16.1.2.1 Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

16.1.2.2 The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

16.1.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

16.1.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

16.1.2.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

16.2 Quantitative analysis:

16.2.1 When a compound has been identified, quantitation of that compound is performed using the area of the quantitation ions listed in Tables 1 and 1A. Quantitation is performed using the internal standard technique. Internal standards are specified in Tables 1 and 1A. For targets not in either Table, the internal standard nearest the retention time of the analyte will be used. All qualitative results will be reported without correction for recovery data, and without subtraction of blank amounts.

16.2.2 Calculate the concentration of each identified analyte in the sample as follows:

Water:

$$\text{Concentration in ug/L} = \frac{(A_x)(I_s)(V_t)(\text{Dil})}{(A_{is})(RF)(V_o)}$$

where:

A_x = Area of characteristic ion for compound being measured
I_s = Amount of internal standard injected in ug/ml
V_t = Volume of total extract in ml
A_{is} = Area of characteristic ion for the internal standard
RF = Average response factor for compound being measured (section 14.5.2)
V_o = Volume of water extracted in liters
Dil = Any post extraction dilution factor

Sediment/Soil Sludge:

$$\text{Concentration in mg/kg} = \frac{(A_x)(I_s)(V_t)(\text{Dil})}{(A_{is})(RF)(W_s)(D)}$$

where:

W_S = Weight of sample extracted in grams
D = % solids
all others same as for water

Note: If the compound detected has not met the 15 percent RSD requirement, the analyst must use the regression line fitted from the initial calibration to determine the extract concentration.

16.2.3 When requested, an estimate of concentration for non calibrated components in the sample is made. The formulas given above are used with the following modifications: The areas A_x and A_{is} are from the total ion chromatograms and the RF for the compound is assumed to be 1. The nearest internal standard free of interferences will be used. The concentration obtained is reported indicating that the value is estimated.

16.2.4 If the response for any quantitation ion exceeds the curve range of the initial calibration of the GC/MS system, a dilution of the extract is made. Dilute the sample only enough to bring the analytes in question into the upper half of the calibration curve. Additional internal standard is added to the diluted extract to maintain the required 5 ng/uL of each internal standard in the extracted volume. The diluted extract is then re-analyzed.

17.0 DATA REPORTING AND DELIVERABLES

17.1 The analyst running a set of samples is also responsible for correctly filling out, turning in, and filing the associated paperwork. It is essential to perform these tasks for the laboratory to be able to provide the client with defensible data.

17.2 LIMS Reporting

17.2.1 When the analyst has finished running a set of samples the data must be turned into the Laboratory Information Management System (LIMS). The benchsheets must be filled in completely to ensure that the results are reported correctly, and the data is associated with the right quality control batch. It is important that all required benchsheet information be filled in correctly and completely. This includes the quality control batch number from the extraction summary, the analytical batch information from the 12 hour shift, and the dilution factor. Dilution Factors are used to elevate the quantitation limits based on post extraction sample dilutions. The dilution factor may or may not equal the dilution ratio made.

17.2.2 Positive results must be recorded by crossing out the printed quantitation limit and recording the positive result on the right hand side. This is the result that has already been through all necessary calculations, including the dilution factor. If a positive result is detected in a diluted sample, the elevated quantitation limit must also be written on the left of the default quantitation limit.

17.2.3 Surrogate results are reported as amount found in the extract, amount spiked in the extract, and percent recoveries. Figure 3 provides an example of a completed analytical benchsheet.

17.2.4 If there are spikes, quality control benchsheets must also be completed and turned in. In addition to the requirements listed elsewhere in this section, quality control benchsheets also require the spiked amounts, percent recoveries, and percent differences where applicable. If out of control results are present due to extraction or severe matrix problems, the exclude (EXC) box must be checked to prevent the data from biasing the recovery window statistics. Figures 4-6 provide examples of quality control benchsheets.

17.2.5 An extracted Method Preparation Blank (MPB) benchsheet must be turned in for every extraction batch. A daily blank (BLK) must be turned in with every 12 hour shift. When the MPB is run, provided the MPB is clean, the MPB may also be the BLK. Turn in both sets of benchsheets. If running both 8270 and 625 samples, BLK, MPB, and Laboratory Fortified Blank (LFB) benchsheets must be turned in for each method. It is important to remember that a LFB cannot be turned in without also having turned in the associated MPB.

17.2.6 If internal chain-of-custody is required it is very important that the COC sheet is filled out correctly and returned to the COC file location.

17.2.7 All LIMS benchsheets (including the COC sheets) are placed in the correctly colored folders and turned into the data entry person responsible for entering the results. The blue folders containing the BLKs should also have the raw data quantitation reports and chromatograms included.

17.3 Laboratory Required Paperwork

17.3.1 All run, maintenance, tape, and standard logbooks must be filled in completely and correctly. Corrections are to be made with a lineout, not a writeover, and must be dated and initialized. Blank lines in the run logbook must be Z'd out. See Figure 7 and 8 for standard and run logbook examples.

17.3.2 All tune, curves and continuing calibration standard runs, and internal standard reports must be placed in their correct binders.

17.3.3 All LIMS documentation (except for the sample and quality control benchsheets), and the raw data (except for that specified above) must be placed in the correct folder and turned into the Semi-Volatile Laboratory technician. The technician will then record the date, time, and contents that were turned in. Sample and quality control benchsheets are returned to the proper folder after data review and approval.

17.4 CLP Like Deliverables

17.4.1 The following copies are required for all data packages that require CLP like deliverables:

- Copies of all tunes including the EICP that has the ion peaks for DFTPP, Benzidine, and Pentachlorophenol with the tailing factor calculations and results, and the ion abundance results and requirements.
- Copies of all associated curves, including the raw data quantitation reports, chromatograms, and Form VIs.
- Copies of all associated continuing calibration standards, including the raw data quantitation reports, chromatograms, and Form VIIs.
- Copies of all sample raw data quantitation reports, including triple plots for positive hits, chromatograms, and all associated TIC reports when requested. Quality control raw data, including blanks, are not necessary.
- Internal standard area count results and requirements for all samples.

17.5 Rounding and Significant Figures

17.5.1 Rounding is performed on the final quantitated result only. For all non-quality control results, results of < 10 ppb are rounded to 1 significant figure. Results ≥ 10 ppb are reported to 2 significant figures.

17.5.2 Quality control results are reported to 3 significant figures.

18.0 QUALITY ASSURANCE

18.1 Each laboratory that uses these methods is required to operate a formal quality control program. The program consist of the following requirements previously covered:

a) DFTPP (section 14.3)
b) Initial Calibration (section 14.5)
c) Continuing Calibration (section 14.6)

18.2 In addition to the above, the following quality control requirements must be followed.

18.3 Blank

18.3.1 After DFTPP and the curve or continuing calibration has been run, a blank is required before any samples are analyzed showing that the analytical system is free from interferences and contamination. This blank will be either a Method Preparation Blank (MPB) or a solvent Daily Blank (BLK). The MPB is the blank extracted with a set of samples, and the BLK is methylene chloride spiked with internal standard. The MPB will be run when available, and the BLK will be run when the extracted MPB has already been run. See section 17.2.3 for blank reporting requirements. At a minimum a blank is run at this point during every 12 hour shift. A blank will be run more frequently if contamination is suspected from a high level sample or if laboratory contamination is in question. MPBs must be spiked with surrogates and carried through the same stages of preparation as the samples.

18.4 Internal Standards

18.4.1 The internal standard responses and retention times in all runs following the continuing calibration standard must be evaluated immediately after or during data acquisition. The retention time for all internal standards must be within ± 30 seconds from the current 12 hour continuing calibration standard. The area of the quantitation ion for all internal standards must stay within a factor of two (-50% to +100%) from the current 12 hour continuing calibration standard.

18.4.2 Any time an internal standard fails the -50% to +100% area criteria, the ability to accurately quantitate an analyte is lost. For that reason every effort will be made to prevent the failure of an internal standard. Refer to the Corrective Action SOP for the procedure to follow when an internal standard fails. If many samples are out of control for no apparent reason, the mass spectrometer needs to be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning are necessary.

18.5 Surrogates

18.5.1 All samples must be spiked with surrogate standards. Until thirty samples of a given matrix have been analyzed, default recovery windows of 50-150% will be used. Once thirty samples of a given matrix have been analyzed, in house recovery windows will be generated (section 18.5.2-18.5.4) and used. Table 6 also provides examples of in house generated surrogate

recovery windows. These are examples only. At a minimum surrogate recovery limits will be updated annually on a matrix by matrix basis.

18.5.2 Calculate the upper and lower control limit for each surrogate standard. This should be done as follows:

Upper Control Limit (UCL) = p + 3s
Lower Control Limit (LCL) = p - 3s

where

p = average percent recovery
s = standard deviation of the percent recovery

18.5.3 Two standard deviations will be used when 3 standard deviations give a negative lower control limit.

18.5.4 If recovery is not within limits, the Corrective Action SOP outlines if, or how the data is to be qualified. If many samples are out of control for no apparent reason, the mass spectrometer needs to be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning are required.

18.6 Matrix and Method Spikes

18.6.1 To assess accuracy, the laboratory must extract a Matrix Spike (SPK1), a Matrix Spike Duplicate (SPK2), and a Laboratory Fortified Blank (LFB), containing the compounds in Table 7, at least once every 20 samples extracted per matrix. If only one to ten samples are analyzed in a month, at least one matrix spike, one matrix spike duplicate, and one laboratory fortified blank is required. The LFB must be extracted daily. Generally the MPB and LFB are run at the beginning of the 12 hour shift, and the matrix spikes at the end. Running the matrix spikes at the end of the shift will help to document that the instrument was still operating correctly during the end of the 12 hour shift.

Note: For the State of Wisconsin samples, all routine 8270C analytes must be added for all spikes and spike duplicates.

18.6.2 Until 5 matrix spikes, matrix spike duplicates, and laboratory fortified blanks have been analyzed, the recoveries of the matrix spike, matrix spike duplicate, and laboratory fortified blank will be validated against default recovery windows of 50-150. Matrix Spike/Matrix Spike Duplicate precision limits will use a default precision limit of ± 20%.

18.6.3 Once five sets of the quality controls samples of a given matrix (waters, soils, etc.) have been analyzed, calculate the average percent recovery (R) and the standard deviation of the percent recovery (SD) per matrix. Express the accuracy assessment as a percent recovery interval from R - 3SD to R + 3SD. If, for example, R = 90 percent and SD = 10 percent, then the accuracy

interval is expressed as 60-120. Another set of limits will be calculated the same way for the laboratory fortified blank windows. Precision windows will be calculated by taking the average of the 5 percent differences ± 3 standard deviations. Windows will be updated at least annually.

18.6.4 Calculate the percent recovery as follows:

$$\%\ \text{recovery} = \frac{(A_{spk} - A_{smp})}{SPK} \times 100$$

where:

Aspk = actual concentration found in the spiked sample, in ug/L
Asmp = amount found in non-spiked sample, in ug/L

$$SPK = \left(\frac{\text{concentration of spiked standard in ug/L} \times \text{ml spiked}}{\text{initial sample volume in L}} \right)$$

18.6.5 Calculate the relative percent difference as follows:

$$\%RPD = \frac{(SPK_1 - SPK_2)}{\left[\frac{(SPK_1 + SPK_2)}{2}\right]} \times 100$$

where:

SPK1 = ug/L found in matrix spike
SPK2 = ug/L found in matrix spike duplicate

18.6.6 If recovery, or precision, is not within limits, the Corrective Action SOP will determine if, or how, the data is to be qualified.

18.6.7 If the LFB is out of control, the problem must be immediately identified and corrected before any further samples for that analyte can be run. The purpose of the LFB is to verify that out-of-control compounds in the MS or MSD are the result of matrix interferences rather than extraction or system errors. Failure of any part of an LFB will require some sort of corrective action, depending on the failed parameter. Every effort will be made to determine the reason for the failure (mis-spiked, mis-extracted, etc.) and appropriate actions will be taken. The Corrective Action SOP details how to proceed when a LFB fails

18.7 Method Detection Limit Studies

18.7.1 A Method Detection Limit (MDL) study must be performed for every analyte to be quantitated by method 8270C. MDL studies must be performed on all mass spectrometers involved in analysis of samples by this method. Results obtained for analytes for which an MDL has not

been calculated by this method must be considered estimated. The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the value is above zero. Actual minimum quantitation limits are derived from the MDL study. MDL studies are run for both waters and soils. Minimum quantitation limits are equal to the amount spiked for the MDL study provided that the MDL passed. The quantitation limit actually achieved in any given analysis will vary depending on instrument sensitivity, matrix effects, and dilutions.

18.7.2 The procedure followed for a MDL study is based on the method given in the Federal Register, Part 136, Appendix B, latest revision. All Method 8270 quality control procedures governing the analyses must be followed.

18.7.3 Seven replicate analyses are performed using reagent water spiked with every analyte of interest, at the estimated minimum quantitation limit concentration.

18.7.4 If a blank measurement is required to calculate the measured level of an analyte, seven separate blanks must be run, one after each of the seven MDL samples. The average blank measurement is subtracted from all seven MDL runs.

18.7.5 The standard deviation of the amount found (blank-subtracted if necessary) for each analyte of the seven runs is calculated and multiplied by 3.143. The resulting number is the calculated MDL.

18.7.6 If the amount spiked is $\geq$ the calculated MDL and $\leq$ 5 times the calculated MDL, and there are no 0 percent recoveries in the set of seven, the MDL result is acceptable. If not the MDL must be re-run, only for the compounds that did not pass. If the study needs to be re-run at a different concentration, the entire set of seven needs to be re-run. If the study does not pass due to poor reproducibility on one of the samples, only that run needs to be re-analyzed. All seven runs do not need to be run in the 12 hour same shift.

18.7.7 If at any time the MDL Level is above a client's or state's desired quantitation limit, the "Calculated MDL" may be used for a quantitation limit if it the report is narrated. The narration needs to state that the quantitation limit is a "Calculated MDL" and when the particular compound is spiked at that level it is not observed.

18.7.8 The curve on the instrument being used to conduct the analysis must have as its low point the vial concentration of a sample spiked at the "MDL Level". A curve starting above this number can lead to large potential errors. For example: If the concentration of a sample spiked at the "MDL Level" of 0.10 ug/L is extracted as the MDL was extracted (i.e. 1000 mL to 1 ml, or 1000x concentration), the vial concentration will be 0.1 mg/L and this must also be the concentration of the low curve point. Consequently, assuming the MDL passed, 0.10 ug/L is the minimum quantitation limit reportable without narrating the results with the above mentioned qualifier.

18.7.9 Table 8 shows an example of a MDL spreadsheet used to calculate and verify MDLs and quantitation limits.

19.0 ANALYST CERTIFICATION/METHOD VALIDATION

19.1 Before the analysis of any actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running a one time analyst certification. While the analyst certification is not instrument dependent, this certification is required on every instrument that will be running samples to demonstrate instrument ability to generate acceptable accuracy and precision.

19.2 Prepare the certification samples at 100 ug/L by spiking 1 mL of a 100 ng/uL standard containing all the compounds of interest into 1 liter and extracting down to 10 ml. The spiking standard used must be prepared independently from the standard used for quantitation.

19.3 Analyze the four distilled check samples following the SOP. Calculate the average recovery (x) in ug/L, and the standard deviation of the recovery (s) in ug/L, for each analyte using the four results.

19.3.1 For each analyte x must be in the range of the current LIMS LFB limits and s must be less than or equal to 20. If s and x for all analytes meet the acceptance criteria, the analyst certification is acceptable. The analyst and the system are now permitted to run samples following this SOP. If any individual s exceeds the precision limit or any individual falls outside the range for accuracy, then the system performance is unacceptable for that analyte.

19.3.2 When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst must proceed according to 19.4.

19.4 Locate and correct the source of the problem and repeat the test for all analytes that failed to meet the criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all unacceptable compounds, beginning with section 19.1. Samples may not be analyzed by any analyst, or on any instrument, until the analyst certification has been successfully completed. Copies of the successful analyst certifications/method validations spreadsheet and raw data should be given to the Quality Assurance Manager.

20.0 REFERENCES

20.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update III, Method 8270C, "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 3, December 1996.

20.2 *40 Code of Federal Regulations, most current edition*, Pt. 136, App. A, Method 625-Base/Neutrals and Acids.

20.3 *40 Code of Federal Regulations, most current edition*, Pt. 136, App. B, Definition and Procedure for the Determination of the Method Detection Limit.

21.0 ATTACHMENTS/APPENDICES

21.1 Table 1 – Targets, Retention Times, Quantitation & Secondary Ions

21.2 Table 1A – Additional Targets, Retention Times, Quantitation & Secondary Ions

21.3 Table 2 – Default Quantitation Limits

21.4 Table 2A – Default Quantitation Limits

21.5 Table 3 – DFTPP Key Ions and Ion Abundance Criteria

21.6 Table 4 – SPCC Criteria

21.7 Table 5 – CCC Criteria

21.8 Table 6 – Surrogates and Percent Recovery Windows

21.9 Table 7 – Spiking Compounds and Percent Recovery Windows

21.10 Table 8 – Method Detection Limit Study Example*

21.11 Table 9 – Analyst Certification Example*

21.12 Figure 1 – Tailing Factor of Pentachlorophenol

21.13 Figure 2 – 100 ppm Continuing Calibration Standard Total Ion Chromatogram

21.14 Figure 3 – Completed Analytical Benchsheet

21.15 Figure 4 – 6 Completed Quality Control Benchsheets

21.16 Figure 7 – Standard Logbook

21.17 Figure 8 – Analytical Run Logbook

*Only part of the Table or Benchsheet example has been included.

TABLE 1
TARGETS, RETENTION TIMES, QUANTITATION & SECONDARY IONS

Compound	Retention Time (min.)*	Quantitation Ion	Secondary Ion(s)	Internal Standard
Acenaphthene-d_{10} (I.S.)	17.76	162	162, 160	3
Chrysene-d_{12} (I.S.)	25.04	240	120, 236	5
Dichlorobenzene-1,4-d_4 (I.S.)	10.06	152	150, 115	1
Naphthalene-d_8 (I.S.)	13.21	136	68	2
Perylene-d_{12} (I.S.)	29.83	264+265	260, 265	6
Phenanthrene-d_{10} (I.S.)	20.50	188	94,80	4
2-Fluorobiphenyl (surr.)	16.00	172	171	3
2-Fluorophenol (surr.)	7.18	112	64	1
Nitrobenzene-d_5 (surr.)	11.38	127+128	128, 54	2
Phenol-d_6 (surr.)	9.13	99		1
2,4,6-Tribromophenol (surr.)	19.33	330	332, 141	3
Terphenyl-o (surr.)	21.03	230	215,229	5
Acenaphthene	17.84	152+154	153, 152	3
Acenaphthylene	17.44	152	151, 153	3
Anthracene	20.63	178	176, 179	4
Benzoic acid	12.39	105	77,122	2
Benzo(a)anthracene	25.00	228	229, 226	5
Benzo(b and k)fluoranthene	28.24/28.40	252	253, 125	6
Benzo(g,h,i)perylene	37.32	276+277	138, 277	6
Benzo(a)pyrene	29.60	252	253, 125	6
Benzyl alcohol	10.35	108	79, 77	1
Bis(2-chloroethoxy)methane	12.51	92+93	95, 123	2
Bis(2-chloroethyl)ether	9.38	92+93	63, 951	1
Bis(2-chloroisopropyl)ether	10.65	45	77, 121	1
Bis(2-ethylhexyl)phthalate	24.59	149	167, 279	5
4-Bromophenyl phenyl ether	19.73	248	250, 141	4
Butyl benzyl phthalate	23.56	149	91, 206	5
4-Chloroaniline	13.40	127	129	2
2-Chloronaphthalene	16.34	162	127, 164	3
4-Chloro-3-methylphenol	14.60	107	144, 142	2
2-Chlorophenol	9.57	128	64, 130	1
4-Chlorophenyl phenyl ether	18.87	204	206, 141	3

Approved By: [signature] 12/14/98
QA Manager

Approved By: [signature] 12/14/98
Area Manager

TABLE 1 (con't)
TARGETS, RETENTION TIMES, QUANTITATION & SECONDARY IONS

Compound	Retention Time (min.)*	Quantitation Ion	Secondary Ion(s)	Internal Standard
Chrysene	25.10	228	226, 229	5
Dibenz(a,h)anthracene	35.57	278	139, 279	6
Dibenzofuran	18.20	168	139	3
Di-n-butylphthalate	21.23	149	150, 104	4
1,3-Dichlorobenzene	9.93	146	148, 111	1
1,4-Dichlorobenzene	10.10	146	148, 111	1
1,2-Dichlorobenzene	10.45	146	148, 111	1
3,3'-Dichlorobenzidine	24.80	252	254, 126	5
2,4-Dichlorophenol	12.84	162	164, 98	2
Diethylphthalate	18.59	149+177	177, 150	3
2,4-Dimethylphenol	12.26	107	121, 122	2
Dimethylphthalate	17.03	163	194, 164	3
4,6-Dinitro-2-methylphenol	18.94	198	51, 105	4
2,4-Dinitrophenol	17.86	184	63, 154	3
2,4-Dinitrotoluene	18.12	165	63, 89	3
2,6-Dinitrotoluene	17.20	165	63, 89	3
1,2-Diphenylhydrazine	19.15	77	105, 182	4
Di-n-octylphthalate	26.32	149	167, 43	6
Fluoranthene	22.35	202	101, 203	4
Fluorene	18.89	166	165, 167	3
Hexachlorobenzene	19.87	284	142, 249	4
Hexachlorobutadiene	13.54	225	223, 227	2
Hexachlorocyclopentadiene	15.45	237	235, 272	3
Hexachloroethane	11.30	119	201, 199	1
Indeno(1,2,3-cd)pyrene	35.52	276+277	138, 227	6
Isophorone	12.00	82	95, 138	2
2-Methylnapthalene	15.06	142	141	2
2-Methylphenol (o-cresol)	10.58	108	79,107	1
3- and/or 4-Methylphenol (m,p-cresol)	10.95	108	79,107	1
Naphthalene	13.26	128	129, 127	2

Approved By: RT 12/14/98 QA Manager
Approved By: JM 12/14/98 Area Manager

TABLE 1 (con't)
TARGETS, RETENTION TIMES, QUANTITATION & SECONDARY IONS

Compound	Retention Time (min.)*	Quantitation Ion	Secondary Ion(s)	Internal Standard
2-Nitroaniline	16.59	138	92, 138	3
3-Nitroaniline	17.62	138	108, 92	3
4-Nitroaniline	18.90	108+138	108, 92	3
Nitrobenzene	11.43	77+123	65	2
2-Nitrophenol	12.21	109+139	109, 65	2
4-Nitrophenol	18.02	65	109, 65	3
N-Nitrosodimethylamine	4.58	73+74	42	1
N-Nitrosodiphenylamine	19.08	169	168, 167	4
N-Nitrosodipropylamine	10.95	70	42, 101, 1	1
Pentachlorophenol	20.18	266	264, 268	4
Phenanthrene	20.54	178	179, 176	4
Phenol	9.17	94	65, 66	1
Pyrene	22.74	202	200, 203	5
1,2,4-Trichlorobenzene	13.05	180	182, 145	2
2,4,5-Trichlorophenol	15.91	196	198, 200	3
2,4,6-Trichlorophenol	15.79	196	198, 200	3

I.S. = Internal Standard
surr. = Surrogate
* retention time provided for example only.

Approved By: [signature] 12/14/98 QA Manager
Approved By: [signature] 12/14/98 Area Manager

TABLE 1A
ADDITIONAL TARGETS, RETENTION TIMES, QUANTITATION & SECONDARY IONS

Compound	Retention Time (min.)*	Quantitation Ion	Secondary Ion(s)	Internal Standard
Acenaphthene-d_{10} (I.S.)	17.05	162	160,164	3
Chrysene-d_{12} (I.S.)	24.14	240	120,236	5
Dichlorobenzene-1,4-d_4 (I.S.)	9.39	152	150,115	1
Naphthalene-d_8 (I.S.)	12.49	136	68	2
Perylene-d_{12} (I.S.)	28.22	264+265	260	6
Phenanthrene-d_{10} (I.S.)	19.96	188	94,80	4
Acetophenone	10.45	105	77,70	2
2-Acetylaminofluorene	23.74	181	223,180	3
4-Aminobiphenyl	18.63	169	168,167	3
Aniline	8.76	93	66,65	1
Aramite**	22.64/22.73/22.85	185	63,57	4
2-sec-Butyl-4,6-dinitrophenol	19.89	211	163,147	3
2,6-Dichlorophenol	12.69	162	164,63	2
p-(Dimethylamino)azobenzene	22.71	120	225,77	3
7,12-Dimethylbenz(a)anthracene	26.79	256	252,241	5
3,3'-Dimethylbenzidine	23.26	212	213,106	3
2,2-Dimethylphenethylamine	12.64	58	91	2
1,3-Dinitrobenzene	16.55	167+168	139,169	2
Ethyl methacrylate	4.98	69	41,99	1
Ethyl methanesulfonate	7.74	79	109,97	1
Hexachlorophene	27.48	196	198,209	6
Hexachloropropene	12.75	213	215,211	2
Isosafrole	15.58	162	131,104	2
Methapyrilene	21.39	58	97,72	3
3-Methylcholanthrene	29.28	268	126,252	5
Methyl methacrylate	3.67	69	41,100	1
Methyl methanesulfonate	6.34	80	65,79	1
1,4-Naphthoquinone	16.23	158	104,102	2
1-Naphthylamine	17.87	143	115,116	2
2-Naphthylamine	18.05	143	115,116	2
4-Nitroquinoline-1-oxide	21.38	190	89,101	4

Approved By: [signature] 12/14/98 QA Manager

Approved By: [signature] 12/14/98 Area Manager

TABLE 1A (con't)
ADDITIONAL TARGETS, RETENTION TIMES, QUANTITATION & SECONDARY IONS

Compound	Retention Time (min.)*	Quantitation Ion	Secondary Ion(s)	Internal Standard
N-Nitroso-di-n-butylamine	13.64	84	57,41	2
N-Nitrosodiethylamine	7.14	102	42,44	1
N-Nitrosomethylethylamine	5.75	88	42,43	1
N-Nitrosomorpholine	10.48	56	116,86	2
N-Nitrosopiperidine	11.24	114	42,56	2
N-Nitrosopyrrolidine	10.39	100	41,42	1
5-Nitro-o-toluidine	18.42	152	106,77	3
Pentachlorobenzene	17.46	250	252,248	3
Pentachloroethane	8.74	167	93,117	2
Pentachloronitrobenzene	19.78	237	169,142	4
Phenacetin	19.17	108	109,179	3
p-Phenylenediamine	13.80	108	80,107	2
2-Picoline	6.73	93	66,92	1
Pronamide	20.20	173	175,145	3
Pyridine	6.32	79	52,51	1
Safrole	14.24	162	131,101	2
1,2,4,5-Tetrachlorobenzene	14.76	216	214,218	3
2,3,4,6-Tetrachlorophenol	17.83	232	230,234	3
o-Toluidine	10.54	107	106,79	2
sym-Trinitrobenzene	19.09	75	74,213	3
Octachlorocyclopentene (C-58)	19.03	307+309	237	3

I.S. = Internal Standard
* retention time provided for example only.
** Aramite chromatographs into three peaks

Approved By: RT 12/14/98 QA Manager
Approved By: JD 12/14/98 Area Manager

TABLE 2
DEFAULT QUANTITATION LIMITS

Semivolatiles	CAS Number	Default Quantitation Limits** Surface/ Ground Water ug/L	Low Soil/ Sediment mg/kg
Acenaphthene	83-32-9	5	0.33
Acenaphthylene	208-96-8	5	0.33
Anthracene	120-12-7	5	0.33
Benzoic acid	65-85-0	50	3.3
Benzo(a)anthracene	56-55-3	5	0.33
Benzo(b and k)fluoranthene	205-99-2 / 207-08-9	5	0.33
Benzo(g,h,i)perylene	191-24-2	5	0.33
Benzo(a)pyrene	50-32-8	5	0.33
Benzyl alcohol	100-51-6	50	1.3
Bis(2-chloroethoxy)methane	111-91-1	5	0.33
Bis(2-chloroethyl)ether	111-44-4	5	0.33
Bis(2-chloroisopropyl)ether	39638-32-9	5	0.33
Bis(2-ethylhexyl)phthalate	117-81-7	5	0.33
4-Bromophenyl phenyl ether	101-55-3	5	0.33
Butyl benzyl phthalate	85-68-7	5	0.33
4-Chloroaniline	106-47-8	20	1.3
2-Chloronaphthalene	91-58-7	5	0.33
4-Chloro-3-methylphenol	59-50-7	5	0.33
2-Chlorophenol	95-57-8	5	0.33
4-Chlorophenyl phenyl ether	7005-72-3	5	0.33
Chrysene	218-01-9	5	0.33
Dibenz(a,h)anthracene	53-70-3	5	0.33
Dibenzofuran	132-64-9	5	0.33
Di-n-butylphthalate	84-74-2	5	0.33
1,3-Dichlorobenzene	541-73-1	5	0.33
1,4-Dichlorobenzene	106-46-7	5	0.33
1,2-Dichlorobenzene	95-50-1	5	0.33
3,3'-Dichlorobenzidine	91-94-1	20	2.0
2,4-Dichlorophenol	120-83-2	5	0.33
Diethylphthalate	84-66-2	5	0.33

Approved By: RL 12/14/98 QA Manager
Approved By: JD 12/14/98 Area Manager

TABLE 2 (con't)
DEFAULT QUANTITATION LIMITS

Semivolatiles	CAS Number	Default Quantitation Limits** Surface/ Ground Water ug/L	Low Soil/ Sediment mg/kg
2,4-Dimethylphenol	105-67-9	5	0.33
Dimethylphthalate	131-11-3	5	0.33
4,6-Dinitro-2-methylphenol	534-52-1	20	1.7
2,4-Dinitrophenol	51-28-5	20	1.7
2,4-Dinitrotoluene	121-14-2	5	0.33
2,6-Dinitrotoluene	606-20-2	5	0.33
Diphenylamine	122-39-4	5	0.33
Di-n-octylphthalate	117-84-0	5	0.33
Fluoranthene	206-44-0	5	0.33
Fluorene	86-73-7	5	0.33
Hexachlorobenzene	118-74-1	5	0.33
Hexachlorobutadiene	87-68-3	5	0.33
Hexachlorocyclopentadiene	77-47-4	5	0.33
Hexachloroethane	67-72-1	5	0.33
Indeno(1,2,3-cd)pyrene	193-39-5	5	0.33
Isophorone	78-59-1	5	0.33
2-Methylnaphthalene	91-57-6	5	0.33
2-Methylphenol (o-cresol)	95-48-7	5	0.33
3- and/or 4-Methylphenol (m,p-cresol)	106-44-5	5	0.33
Naphthalene	91-20-3	5	0.33
2-Nitroaniline	88-74-4	20	1.7
3-Nitroaniline	99-09-2	20	1.7
4-Nitroaniline	100-01-6	20	1.7
Nitrobenzene	98-95-3	5	0.33
2-Nitrophenol	88-75-5	5	0.33
4-Nitrophenol	100-02-7	20	1.7
N-Nitrosodimethylamine	62-75-9	5	0.33
N-Nitrosodiphenylamine	86-30-6	5	0.33
N-Nitrosodipropylamine	621-64-7	5	0.33
Pentachlorophenol	87-86-5	20	1.7

Approved By: RL 12/14/98 QA Manager
Approved By: JD 12/14/98 Area Manager

TABLE 2 (con't)
DEFAULT QUANTITATION LIMITS

Semivolatiles	CAS Number	Default Quantitation Limits** Surface/ Ground Water ug/L	Low Soil/ Sediment mg/kg
Phenanthrene	85-01-8	5	0.33
Phenol	108-95-2	5	0.33
Pyrene	129-00-0	5	0.33
1,2,4-Trichlorobenzene	120-82-1	5	0.33
2,4,5-Trichlorophenol	95-95-4	5	0.33
2,4,6-Trichlorophenol	88-06-2	5	0.33

* Sample quantitation limits are highly matrix-dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

** Water quantitation limits are based on 1000 mL to 1 mL extracted MDL study. Quantitation limits listed for soil/sediment are based on an extraction of 30 g to 1 ml. The sample must be ≥ 50% solids.

Approved By: RL 12/14/98 QA Manager
Approved By: JO 12/14/98 Area Manager

TABLE 2A
ADDITIONAL DEFAULT QUANTITATION LIMITS

Semivolatiles	CAS Number	Default Quantitation Limits** Surface/ Ground Water ug/L	Low Soil/ Sediment mg/kg
Acetophenone	98-86-2	10	0.33
2-Acetylaminofluorene	53-96-3	50	2.0
4-Aminobiphenyl	92-67-1	10	0.33
Aniline	62-53-3	5	1.7
Aramite	140-57-8	50	2.0
2-sec-Butyl-4,6-dinitrophenol	88-85-7	5	2.0
2,6-Dichlorophenol	87-65-0	5	0.33
p-(Dimethylamino)azobenzene	60-11-7	10	0.33
7,12-Dimethylbenz(a)anthracene	57-97-6	10	0.33
3,3'-Dimethylbenzidine	119-93-7	50	2.0
2,2-Dimethylphenethylamine	122-09-8	20	0.70
1,3-Dinitrobenzene	99-65-0	5	0.33
Ethyl methacrylate	97-63-2	50	2.0
Ethyl methanesulfonate	62-50-0	10	1.0
Hexachlorophene	70-30-4	***	***
Hexachloropropene	1888-71-7	50	2.0
Isosafrole	120-58-1	20	0.70
Methapyrilene	91-80-5	10	1.0
3-Methylcholanthrene	56-49-5	50	2.0
Methyl methacrylate	80-62-6	50	2.0
Methyl methanesulfonate	66-27-3	50	2.0
1,4-Naphthoquinone	130-15-4	1000	30
1-Naphthylamine	134-32-7	50	2.0
2-Naphthylamine	91-59-8	50	2.0
4-Nitroquinoline-1-oxide	56-57-5	500	20
N-Nitroso-di-n-butylamine	924-16-3	20	0.90
N-Nitrosodiethylamine	55-18-5	5	2.0
N-Nitrosomethylethylamine	62-75-9	5	2.0
N-Nitrosomorpholine	59-89-2	20	0.70
N-Nitrosopiperidine	100-75-4	20	0.70

Approved By: RL 12/14/98 QA Manager Approved By: JO 12/14/98 Area Manager

TABLE 2A (con't)
ADDITIONAL DEFAULT QUANTITATION LIMITS

Semivolatiles	CAS Number	Default Quantitation Limits** Surface/ Ground Water ug/L	Low Soil/ Sediment mg/kg
N-Nitrosopyrrolidine	930-55-2	20	0.70
5-Nitro-o-toluidine	99-55-8	10	0.33
Pentachlorobenzene	608-93-5	5	0.33
Pentachloroethane	76-01-7	50	2.0
Pentachloronitrobenzene	82-68-8	20	0.50
Phenacetin	62-44-2	10	0.33
p-Phenylenediamine	106-50-3	***	***
2-Picoline	109-06-8	20	0.70
Pronamide	23950-58-5	10	0.33
Pyridine	110-86-1	10	0.33
Safrole	94-59-7	10	0.33
1,2,4,5-Tetrachlorobenzene	95-94-3	10	0.50
2,3,4,6-Tetrachlorophenol	58-90-2	50	2.0
o-Toluidine	95-53-4	10	0.33
sym-Trinitrobenzene	99-35-4	20	0.70
Octachlorocyclopentene (C-58)	706-78-5	0.1	0.02

* Sample quantitation limits are highly matrix-dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

** Water quantitation limits are based on 1000 mL to 1 mL extracted MDL study. Quantitation limits listed for soil/sediment are based on an extraction of 30 g to 1 ml. The sample must be ≥ 50% solids.

*** These compounds have been demonstrated to be difficult to extract from water, or difficult to chromatograph. Detection limits are not available.

TABLE 3
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30-80% of mass 198
68	<2% of mass 69
69	Present
70	<2% of mass 69
127	25-75% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance (see note)
199	5-9% of mass 198
275	10-30% of mass 198
365	>0.75% of mass 198
441	Present but less than mass 443
442	40-110% of mass 198
443	15-24% of mass 442

NOTE: All ion abundances MUST be normalized to m/z 198, the nominal base peak, even though the ion abundances of m/z 442 may be up to 110% that of m/z 198.

TABLE 4
SYSTEM PERFORMANCE CHECK COMPOUNDS (SPCCs)

Base/Neutral Fraction	Acid Fraction
N-nitroso-di-n-propylamine	2,4-Dinitrophenol
Hexachlorocyclopentadiene	4-Nitrophenol

TABLE 5
CALIBRATION CHECK COMPOUNDS (CCCs)

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitroso-di-n-phenylamine	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

Approved By: RL 12/14/98 QA Manager

Approved By: JO 12/14/98 Area Manager

TABLE 6
EXAMPLE* OF SURROGATE SPIKE RECOVERY LIMITS
FOR WATER AND SOIL/SEDIMENT SAMPLES

Surrogate Compound	Calculated % Recoveries Water	Calculated % Recoveries Soil
Nitrobenzene-d_5	34-120	15-132
2-Fluorobiphenyl	39-118	30-124
o-Terphenyl	29-133	26-128
Phenol-d_6	2-46	12-133
2-Fluorophenol	6-69	3-129
2,4,6-Tribromophenol	28-130	5-132

* This Table is provided as an example only. Actual windows are updated periodically and may be different at the time samples are run.

Approved By: 12/14/98 QA Manager Approved By: 12/14/98 Area Manager

TABLE 7
EXAMPLE* OF MATRIX SPIKE, MATRIX SPIKE DUPLICATE, AND LABORATORY FORTIFIED BLANK ACCEPTABLE RECOVERY AND PERCENT DIFFERENCE WINDOWS

SPIKED COMPOUND WINDOWS	WATER MS % RECOVERY WINDOWS	WATER PRECISION WINDOWS	WATER LFB % RECOVERY WINDOWS	SOIL MS % RECOVERY WINDOWS	SOIL PRECISION WINDOWS	SOIL LFB % RECOVERY WINDOWS
Acenaphthene	36-117	15	37-116	30-117	17	32-110
4-Chloro-3-methylphenol	43-117	19	46-117	40-126	21	44-113
2-Chlorophenol	39-116	22	43-117	44-119	20	49-112
1,4-Dichlorobenzene	43-118	22	43-116	42-112	35	45-108
2,4-Dinitrotoluene	45-116	18	46-115	34-123	19	37-114
N-nitroso-di-n-propylamine	38-128	20	47-118	38-124	22	48-110
Naphthalene	40-124	17	55-115	46-122	20	51-111
4-Nitrophenol	1-69	53	4-55	21-129	33	34-113
Pentachlorophenol1	1-140	24	17-132	1-129	36	15-118
Phenol	2-60	44	9-55	34-134	23	39-126
Pyrene	35-129	18	39-123	37-122	21	36-114
1,2,4-Trichlorobenzene	43-113	21	47-109	42-114	23	46-107

* This Table is provided as an example only. Actual windows are updated periodically and may be different at the time samples are run.

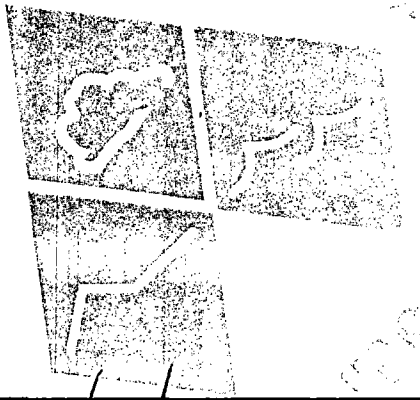


TABLE 8

METHOD DETECTION LIMIT STUDY EXAMPLE*

Parameter / Compound	Date Analyzed	Reference Citation	Units	Amount Spiked	Rep. #1	Rep. #2	Rep. #3	Rep. #4	Rep. #5	Rep. #6	Rep. #7	Average Amount Found	Average Percent Recovery	Standard Deviation	Calculated MDL
Pyridine	3/20/98	8270	ug/L	0.100	0.032	0.009	0.017	0.013	0.029	0.026	0.020	0.021	21%	0.009	0.027
Phenol	3/20/98	8270	ug/L	0.100	0.033	0.049	0.044	0.050	0.037	0.027	0.034	0.039	39%	0.009	0.027
Aniline	3/20/98	8270	ug/L	0.100	0.028	0.021	0.012	0.014	0.013	0.021	0.020	0.018	18%	0.006	0.018
Bis(2-Chloroethyl)Ether	3/20/98	8270	ug/L	0.100	0.091	0.080	0.079	0.088	0.091	0.109	0.090	0.090	90%	0.010	0.031
2-Chlorophenol	3/20/98	8270	ug/L	0.100	0.068	0.081	0.083	0.085	0.078	0.078	0.076	0.078	78%	0.006	0.017
1,3-Dichlorobenzene	3/20/98	8270	ug/L	0.100	0.070	0.077	0.078	0.078	0.080	0.076	0.065	0.075	75%	0.005	0.017
1,4-Dichlorobenzene	3/20/98	8270	ug/L	0.100	0.072	0.077	0.076	0.074	0.078	0.073	0.065	0.074	74%	0.004	0.014
Benzyl Alcohol	3/20/98	8270	ug/L	0.100	0.075	0.062	0.078	0.089	0.058	0.089	0.105	0.079	79%	0.016	0.052
1,2-Dichlorobenzene	3/20/98	8270	ug/L	0.100	0.066	0.078	0.078	0.073	0.078	0.074	0.062	0.073	73%	0.006	0.020
2-Methylphenol	3/20/98	8270	ug/L	0.100	0.066	0.084	0.077	0.071	0.057	0.049	0.057	0.066	66%	0.012	0.039
Bis(2-Chloroisopropyl)Ether	3/20/98	8270	ug/L	0.100	0.080	0.094	0.054	0.056	0.092	0.089	0.091	0.079	79%	0.017	0.054
4-Methylphenol	3/20/98	8270	ug/L	0.100	0.062	0.088	0.081	0.068	0.048	0.050	0.061	0.065	65%	0.015	0.047
n-Nitroso-di-n-propylamine	3/20/98	8270	ug/L	0.100	0.106	0.106	0.120	0.102	0.110	0.095	0.119	0.108	108%	0.009	0.028
Hexachloroethane	3/20/98	8270	ug/L	0.100	0.090	0.089	0.100	0.097	0.100	0.095	0.082	0.093	93%	0.007	0.021
Nitrobenzene	3/20/98	8270	ug/L	0.100	0.123	0.126	0.113	0.118	0.138	0.141	0.113	0.125	125%	0.011	0.036

* This spreadsheet is provided as an EXAMPLE of the style spreadsheet used for calculating MDLs.

Approved By: RL 12/14/98
QA Manager

Approved By: JC 12/14/98
Area Manager

TABLE 9
Analyst Certification Example*

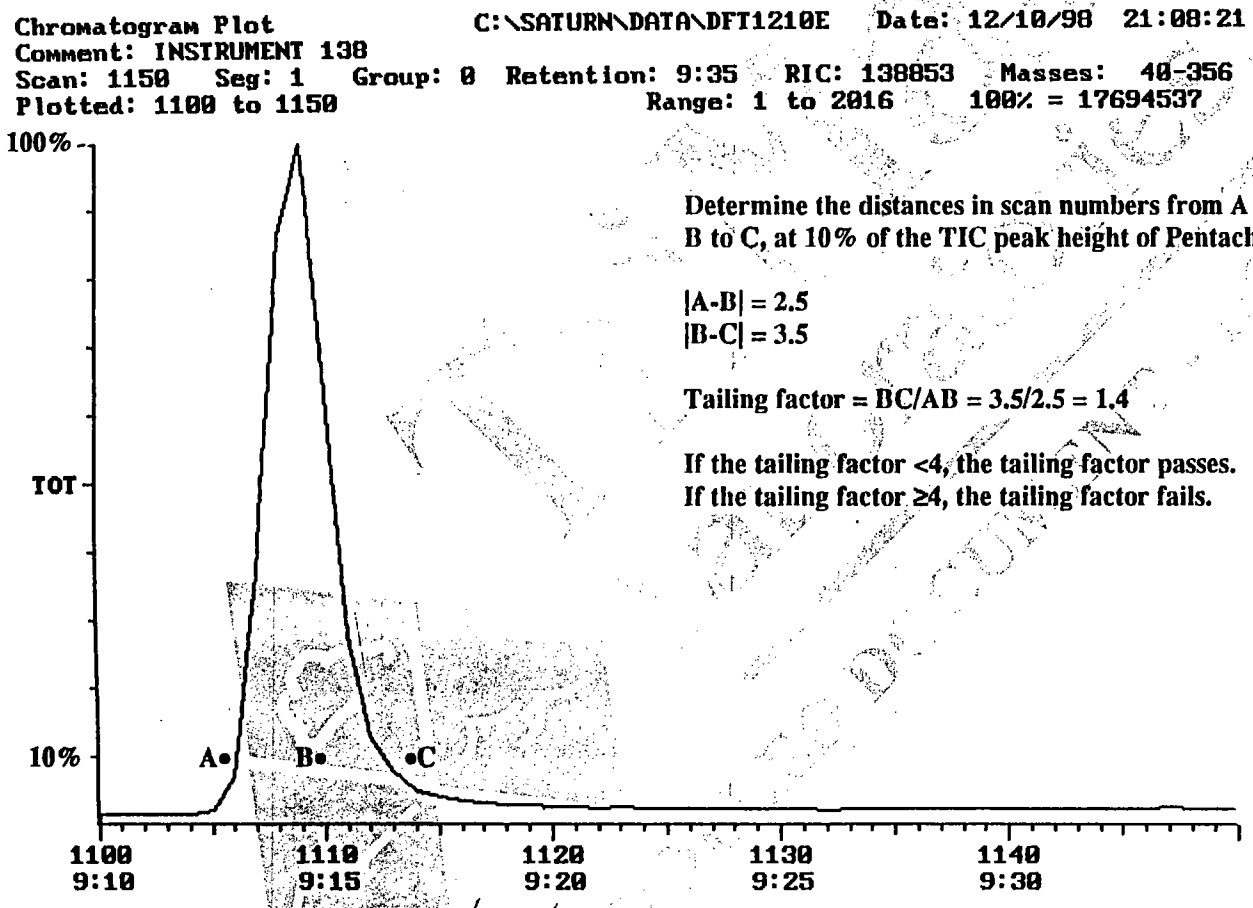
TARGET	AMT SPK ug/L	#1 AMT FND ug/L	#2 AMT FND ug/L	#3 AMT FND ug/L	#4 AMT FND ug/L	AVG REC ug/L	TABLE 6 RECOVERY RANGE in ug/L	PASS/ FAIL REC	STD DEV	STD DEV LIMIT	STD DEV PASS/ FAIL	OVERALL PASS/FAIL
Acenaphthene	100	85.4	90.1	88.0	88.1	87.9	60.1 -132.3	PASS	1.9	27.6	PASS	PASS
Acenaphthylene	100	88.5	91.6	89.8	90.5	90.1	53.5 -126.0	PASS	1.3	40.2	PASS	PASS
Anthracene	100	95.3	99.2	99.7	96.5	97.7	43.4 -118.0	PASS	2.1	32.0	PASS	PASS
Benzo (a) anthracene	100	94.5	97.6	99.3	97.2	97.2	41.8 -133.0	PASS	2.0	27.6	PASS	PASS
Benzo (b) fluoranthene	100	94.5	94.9	97.3	96.2	95.7	42.0 -140.4	PASS	1.3	38.8	PASS	PASS
Benzo (k) fluoranthene	100	94.5	94.9	97.3	96.2	95.7	25.2 -145.7	PASS	1.3	32.3	PASS	PASS
Benzo (a) pyrene	100	93.4	94.5	96.7	95.8	95.1	31.7 -148.0	PASS	1.4	39.0	PASS	PASS
Benzo (g,h,i) perylene	100	96.0	94.4	97.3	97.2	96.2	0.0 -195.0	PASS	1.4	58.9	PASS	PASS
Butyl benzyl phthalate	100	88.9	95.1	92.0	91.4	91.9	0.0 -139.9	PASS	2.5	23.4	PASS	PASS
bis- (2-chloroethyl) ether	100	78.7	85.1	79.0	81.6	81.1	42.9 -126.0	PASS	3.0	55.0	PASS	PASS
bis- (2-chloroethoxy) methane	100	87.1	93.8	86.1	89.2	89.1	49.2 -164.7	PASS	3.4	34.5	PASS	PASS
bis- (2-chloroisopropyl) ether	100	61.7	88.5	80.5	85.6	79.1	62.8 -138.6	PASS	12.0	46.3	PASS	PASS
bis- (2-ethylhexyl) phthalate	100	91.4	97.9	96.7	94.1	95.0	28.9 -136.8	PASS	2.9	41.1	PASS	PASS
Bromophenyl phenyl ether, 4-	100	94.7	99.9	97.7	99.5	98.0	64.9 -114.4	PASS	2.4	23.0	PASS	PASS
Chloronaphthalene, 2-	100	86.2	92.4	87.2	88.2	88.5	64.5 -113.5	PASS	2.7	13.0	PASS	PASS
Chlorophenyl phenyl ether, 4-	100	87.7	93.1	90.9	93.6	91.3	38.4 -144.7	PASS	2.7	33.4	PASS	PASS
Chrysene	100	96.4	102.2	95.1	97.4	97.8	44.1 -139.9	PASS	3.1	48.3	PASS	PASS

* This spreadsheet is provided as an EXAMPLE of the style spreadsheet used for calculating analyst certifications only.

Approved By: _____ 12/14/98
QA Manager

Approved By: _____ 12/14/98
Area Manager

FIGURE #1
TAILING FACTOR OF PENTACHLOROPHENOL



Determine the distances in scan numbers from A to B, and B to C, at 10% of the TIC peak height of Pentachlorophenol.

|A-B| = 2.5
|B-C| = 3.5

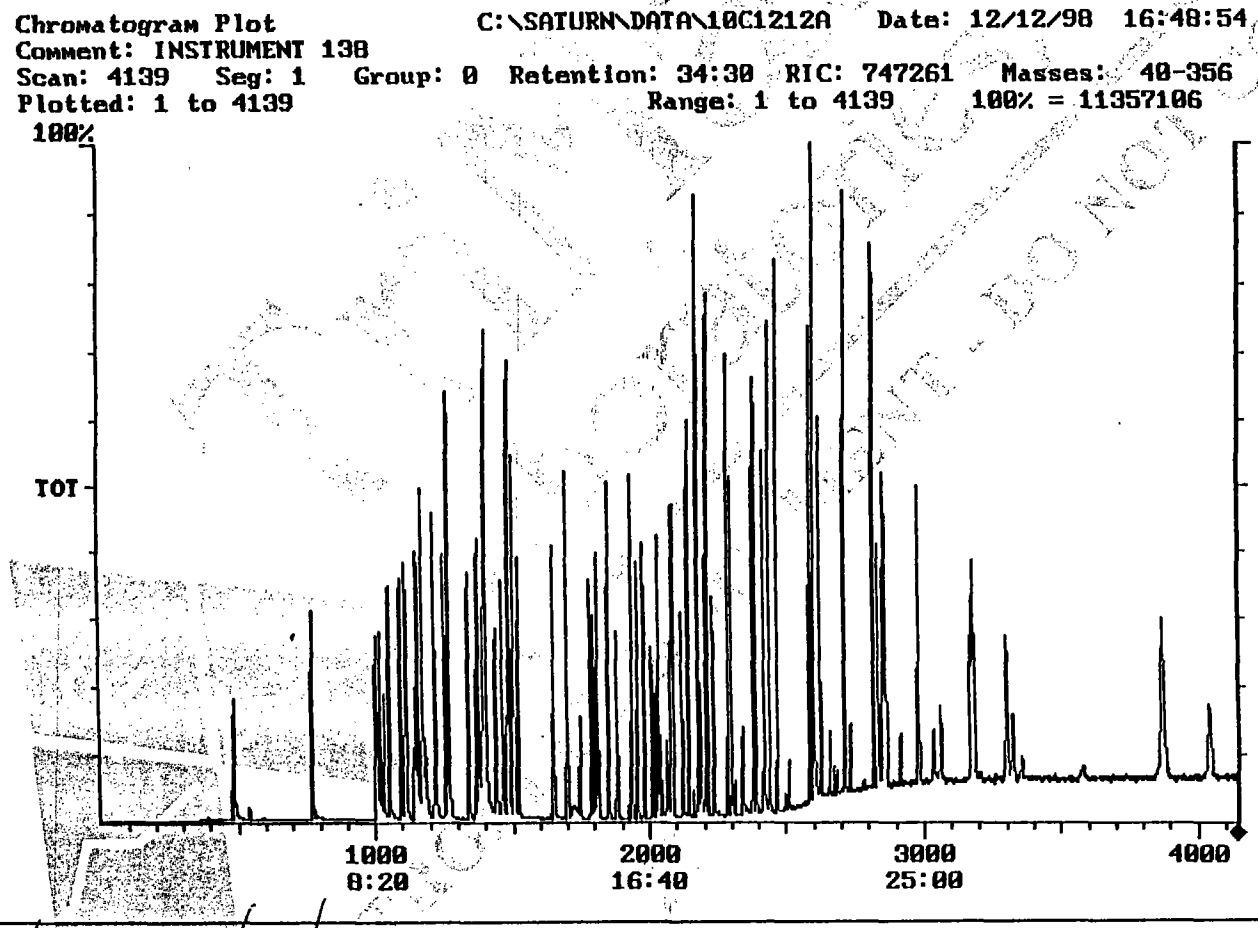
Tailing factor = BC/AB = 3.5/2.5 = 1.4

If the tailing factor <4, the tailing factor passes.
If the tailing factor ≥4, the tailing factor fails.

Approved By: _____ 12/14/98
QA Manager

Approved By: _____ 12/14/98
Area Manager

FIGURE #2
10 ppm BNA 8270C CONTINUING CALIBRATION STANDARD
TOTAL ION CHROMATOGRAM



Approved By: 12/14/98
QA Manager

Approved By: 12/14/98
Area Manager

FIGURE #3
COMPLETED ANALYTICAL BENCHSHEET

24-NOV-98 Type: REG RC1 RC2 RMS FRACTION BENCHSHEET SEMI-VOLATILE LAB PAGE 9

Test N: 1308 1- 50, 2
Parameter: SEMI-VOL'S 8270 TCL LIST
Method: SEMI-VOL/MS/SOL
Ref. cit.: USEPA-8270 Unit: ug/kg dry

Client [redacted]
Project: 34715
Proj Chem: JLR
Submittal: 9 Nov 15-19, 1998 Samples
Sample: 212140 L107C-04

Hold date: 25-DEC-1998
Lab due date: 04-DEC-1998
Client due date: 08-DEC-1998
Received date: 24-NOV-1998

QC: LV5 COC:
C=1 F=1 H=1 R=1

Benchsheet ID: 214616
Analyst: DMA
Date Run: 12-1-98
Instrument N: 11X
Stock Std: SWA1123
Rack#:
Reviewed by: MWL
QC Batch Number: SBC565-123

Initial wt./vol.: 30.2
Final volume: 10.0
% Solids: 78
Dilution Factor: 1
Batch Number:
Batch Owner: JJB
Batch Opened Date: 12-1-98
Batch Seq: 1

	Parameter	ODL/Spk	Result	Percent	LCL	UCL
4	ACENAPHTHENE		CO.33			
5	ACENAPHTHYLENE		CO.33			
12	ANTHRACENE		CO.33			
16	BENZO (A) ANTHRACENE		CO.33			
17	BENZO (A) PYRENE		CO.33			
19	BENZO (B) FLUORANTHENE		CO.33			
20	BENZO (G,H,I) PERYLENE		CO.33			
21	BENZO (K) FLUORANTHENE		CO.33			
26	BIS (2-CHLOROETHYL) ETHER		CO.33			
27	BIS (2-CHLOROISOPROPYL)E		CO.33			
28	BIS (2-ETHYL HEXYL) PHTHA		CO.33			
29	BIS(2CHLOROETHOXY)METHANE		CO.33			
30	4-BROMOPHENYL PHENYL ETHE		CO.33			
32	BUTYL BENZYL PHTHALATE		CO.33			
35	CARBAZOLE		CO.33			
37	4-CHLORO-3-METHYLPHENOL		CO.33			
40	4-CHLOROANILINE		C1.3			
41	2-CHLORONAPHTHALENE		CO.33			
42	2-CHLOROPHENOL		CO.33			
43	4-CHLOROPHENYL PHENYL ETH		CO.33			
44	CHRYSENE		CO.33			
51	DI-N-BUTYL PHTHALATE		CO.33			
52	DI-N-OCTYLPHTHALATE		CO.33			
53	DIBENZO (A,H) ANTHRACENE		CO.33			
54	DIBENZOFURAN		CO.33			
56	1,2-DICHLOROBENZENE		CO.33			
57	1,3-DICHLOROBENZENE		CO.33			
58	1,4-DICHLOROBENZENE		CO.33			
59	3,3'-DICHLOROBENZIDINE		C2			
60	2,4-DICHLOROPHENOL		CO.33			
64	DIETHYLPHTHALATE		CO.33			
66	DIMETHYL PHTHALATE		CO.33			
70	2,4-DIMETHYLPHENOL		CO.33			
71	4,6-DINITRO-2METHYLPHENOL		C1.7			
73	2,4-DINITROPHENOL		C1.7			
74	2,6-DINITROTOLUENE		CO.33			
75	2,4-DINITROTOLUENE		CO.33			
90	FLUORANTHENE		CO.33			

* - Result C corresponding ODL

TRIMATRIX LABORATORIES, INC

Approved By: [signature] 12/14/98
QA Manager

Approved By: [signature] 12/14/98
Area Manager

FIGURE #4
COMPLETED QUALITY CONTROL BENCHSHEET

26-OCT-98 FRACTION BENCHSHEET ## QC ## SEMI-VOLATILE LAB PAGE 9

Test N: 381. 11- 30.02
Parameter: QC SEMI'S 8270-SOIL(FULL)
Method: SEMI-VOL/MS/SOL
Ref. cit.: USEPA-8270 Unit: ug/kg dry

QC Type: MPB

Analyst: MAL
Date run: 12-1-98
Instrument N: 111
Stock std N: SVA1-21-L
Rack N:
Reviewed by: [illegible]
QC Batch Number: 120565-102

Initial wt./vol.: 30.0
Final volume: 10.0
% Solids: —
Dilution factor: 1
Batch Number: 130887
Batch Owner: 111
Batch Opened Date: 12-1-98
Batch Seq: 1

Seq	Parameter	ODL	Orig conc	Result	Spike qty	% Rec	LCL	UCL	EXC
4.	ACENAPHTHENE	0.33			XXXXX				
5.	ACENAPHTHYLENE	0.33			XXXXX				
11.	ANILINE	1.7			XXXXX				
12.	ANTHRACENE	0.33			XXXXX				
15.	BENZIDINE	5			XXXXX				
16.	BENZO (A) ANTHRACENE	0.33			XXXXX				
17.	BENZO (A) PYRENE	0.33			XXXXX				
18.	BENZO (BAK) FLUORANTHENE	0.33			XXXXX				
20.	BENZO (G,H,I) PERYLENE	0.33			XXXXX				
22.	BENZOIC ACID	3.3			XXXXX				
24.	BENZYL ALCOHOL	1.3			XXXXX				
26.	BIS (2-CHLOROETHYL) ETHER	0.33			XXXXX				
27.	BIS (2-CHLOROISOPROPYL)E	0.33			XXXXX				
28.	BIS (2-ETHYL HEXYL) PHTHA	0.33		0.322 J	XXXXX				
29.	BIS(2CHLOROETHOXY)METHANE	0.33			XXXXX				
30.	4-BROMOPHENYL PHENYL ETHE	0.33			XXXXX				
32.	BUTYL BENZYL PHTHALATE	0.33			XXXXX				
37.	4-CHLORO-3-METHYLPHENOL								

- Result < corresponding ODL

TRIMATRIX LABORATORIES, INC.

Approved By: [signature] 12/14/98
QA Manager

Approved By: [signature] 12/14/98
Area Manager

FIGURE #5
COMPLETED QUALITY CONTROL BENCHSHEET

26-OCT-98 FRACTION BENCHSHEET ## QC ## SEMI-VOLATILE LAB PAGE 8

Test #: 381 3- 50.02
Parameter: QC, SEMI'S B/N/A 8270-SOIL
Method: SEMI-VOL/MS/SOL
Ref. cit.: USEPA-8270 Unit: mg/kg dry

QC Type: MPB LFB

Analyst: [illegible]
Date run: 14-J-98
Instrument #: 133
Stock std #: SVA 1.22.C
Rack #: ____
Reviewed by: [illegible]
QC Batch Number: WG569-102

Initial wt./vol.: 30.5 g
Final volume: 10 ml
% Solids: ____
Dilution factor: 1
Batch Number: ____
Batch Owner: 133
Batch Opened Date: 14-J-98
Batch Seq: 1

Seq	Parameter	ODL	MPB Result	SUR (MPB) spike qty	LFB Result	LFB/SUR spike qty	MPB %	LFB %	LFB LCL	LFB UCL	EXC
4.	ACENAPHTHENE	0.33		XXXXX	2.35	3.33		71.8	37	116	
37.	4-CHLORO-3-METHYLPHENOL	0.33		XXXXX	5.10	6.67		76.6	45	118	
43.	2-CHLOROPHENOL	0.33		XXXXX	5.04	"		75.7	46	112	
58.	1,4-DICHLOROBENZENE	0.33		XXXXX	2.58	3.33		77.5	41	107	
75.	2,4-DINITROTOLUENE	0.33		XXXXX	2.67			80.2	40	117	
124	N-NITROSODI-N-PROPYLAMINE	0.33		XXXXX	2.48			74.5	38	114	
132	NAPHTHALENE	0.33		XXXXX	2.80			84.1	45	114	
141	4-NITROPHENOL	1.7		XXXXX	4.31	6.67		64.7	37	121	
159	PENTACHLOROPHENOL	1.7		XXXXX	3.35			50.3	20	126	
163	PHENOL	0.33		XXXXX	4.69			70.4	42	120	
168	PYRENE	0.33		XXXXX	3.36	3.33		70.9	33	131	
182	1,2,4-TRICHLOROBENZENE	0.33		XXXXX	2.87			86.2	40	109	
1.	2-FLUOROBIPHENYL-SUR	XXXXX			80.6	100		81	30	124	
2.	2-FLUOROPHENOL-SUR	XXXXX			145	200		73	3	129	
3.	D5-NITROBENZENE-SUR	XXXXX			63.4	100		63	15	132	
4.	D6-PHENOL-SUR	XXXXX			168	200		84	12	133	
5.	O-TERPHENYL-SUR	XXXXX			71.3	100		72	28	128	
6.	2,4,6-TRIBROMOPHENOL-SUR	XXXXX			185	200		92	5	132	

- Result C corresponding ODL

TRIMATRIX LABORATORIES, INC.

Approved By: [signature] 12/14/98
QA Manager

Approved By: [signature] 12/14/98
Area Manager

FIGURE #6
COMPLETED QUALITY CONTROL BENCHSHEET

31-AUG-98 FRACTION BENCHSHEET ** QC ** SEMI-VOLATILE LAB PAGE 14

Test #: 381 3- 50.02
Parameter: QC SEMI'S B/N/A 8270-SOIL
Method: SEMI-VOL/MS/SOL
Ref. Lit.: USEPA-8270 Unit: ug/kg dry

Analyst: MKI
Date run: 12-3-98
Instrument #: 111
Stock std #: SVH 11.L
Rack #:
Reviewed by:
QC Batch Number: MG568-102

Initial wt./vol.: 30.5 g
Final volume: 10 ml
% Solids: 81
Dilution factor:
Batch Number: 1
Batch Owner: 111
Batch Opened Date: 12-3-98
Batch Seq: 1

Sample Number: 212141 @
QC Type: SPK; MSD Range 1; MSD Range 2
Range 1: SPK1 (= 10 x ODL
Range 2: SPK1) 10 x ODL

Seq	Parameter	ODL	Orig Conc.	SPK1 Result	Spike qty	SPK2 Result	SPK1 %	SPK2 %	SPK LCL	SPK UCL	MSD % Dif	MSD 1 UCL	MSD 2 UCL	EXC
4	ACENAPHTHENE	0.33	X	3.34	3.3	2.16	70.3	64.9	33	132	8.0	16	17	
17	4-CHLORO-3-METHYLPHENOL	0.33		5.33	6.6	5.02	80.0	75.9	43	130	6.0	21	18	
40	2-CHLOROPHENOL	0.33		5.30	"	5.01	79.6	75.2	42	119	5.6	17	20	
50	1,4-DICHLOROBENZENE	0.33		2.60	3.3	2.37	78.1	81.2	37	113	6.3	24	35	
75	2,4-DINITROTOLUENE	0.33		2.66		2.49	79.9	74.8	25	134	6.6	17	19	
124	N-NITROSODI-N-PROPYLAMINE	0.33		2.54		2.58	75.3	77.5	37	121	2.4	22	13	
132	NAPHTHALENE	0.33		2.66		2.65	79.9	79.6	48	120	< 1	20	19	
141	4-NITROPHENOL	1.7		4.98	6.6	4.99	74.8	75.9	33	122	3.9	17	33	
159	PENTACHLOROPHENOL	1.7		3.52		3.52	52.8	52.8	4	139	0	24	35	
163	PHENOL	0.33	X	5.06		4.96	76.0	73.0	35	137	4.0	18	29	
180	PYRENE	0.33	(0.15) J	2.10	3.3	2.50	76.6	70.6	43	128	7.7	16	21	
182	1,2,4-TRICHLOROBENZENE	0.33	X	3.03	"	2.80	91.0	84.1	37	118	7.9	23	12	
1	2-FLUOROBIPHENYL-SUR	XXXXX	XXXXX	81.0	100	81.8	81	84	30	124	XXXXX			
2	2-FLUOROPHENOL-SUR	XXXXX	XXXXX	141	200	139	71	70	3	129	XXXXX			
3	D5-NITROBENZENE-SUR	XXXXX	XXXXX	58.9	100	55.8	59	60	15	132	XXXXX			
4	D6-PHENOL-SUR	XXXXX	XXXXX	178	200	166	89	83	12	133	XXXXX			
5	D-TERPHENYL-SUR	XXXXX	XXXXX	93.0	100	91.0	93	91	28	129	XXXXX			
6	2,4,6-TRIBROMOPHENOL-SUR	XXXXX	XXXXX	202	200	185	101	92	5	132	XXXXX			

* - Result C corresponding ODL

TRIMATRIX LABORATORIES, INC.

Approved By: [signature] 12/14/98
QA Manager

Approved By: [signature] 12/14/98
Area Manager

FIGURE #7
STANDARD LOGBOOK

ORGANICS STOCK STANDARDS

STOCK STD #: SV81,21.0 | SOLVENT USED: MeCl2 | CHECKED TO STD #:
STOCK STD NAME: B/N/A STD. | LOT NUMBER: | CHECKED BY/DATE:
PREPARED BY/DATE: JMH 10/9/98 | FINAL VOLUME: 10ml | USING INSTRUMENT #:
MATH CHECKED BY: | EXPIRATION DATE: 10/9/99 | STORAGE AREA: SV.HLS

ANALYTE, STD # or PURCHASED STD	VENDOR (IF BOUGHT)/ NAME (IF MADE)	LOT #	% PURITY	EXPIRATION DATE	DENSITY or STD CONC	WT /VOL USED	CONCENTRATION
Phenols	Absolute	032796		032601	2000mg/L	1ml	200ppm
PAH mix	↑	010696		010701	↑	↑	↑
Benzidines		052996		053001			
Base/Neutrals Mix #1		082897		082903			
Toxic Substances #1		042096		042101			
Base/Neutrals #2		072196		072201	↓	↓	↓
Toxic Substances #2	Absolute	060696		060703	2000mg/L	1ml	200ppm
B/N SVOC MS1217	In house			060699	4000mg/L	500ml	200ppm
Acid SVOC MS1218	↓			060699	4000mg/L	500ml	200ppm
Pyridine SV81220				100999	2132mg/L	1ml	213ppm

STDPRPOD.XLS | TriMatrix Laboratories, Inc. | page #: 21A of 50

Approved By: [signature] 12/14/98
QA Manager

Approved By: [signature] 12/14/98
Area Manager

FIGURE #8
ANALYTICAL RUN LOGBOOK

Instrument ID: Saturn GC/MS Instrument #133 Date: 12/11/98 Taped: _____ Disk ID: _____ Analyst: MEL

Instrument Settings	Acquisition Information	Temperature Program					Miscellaneous
Multiplier: 2775	Scan Range: 40-450 amu	GC	Temp. (C)	Rate (C/min)	Time (min)	Total (min)	New Column Date: 12/11/98
AGC 20000	Seconds/Scan: 0.500	Step #1	45	0.0	1.5	1.5	New Septa: (Yes) / No
Interface Temp: 250C	Filament Delay: 3.5 min.	Step #2	200	10	15.5	17.0	Working Std. #: SWL122
Source Temp: 190C	Threshold: 2 counts	Step #3	315	25	4.6	21.6	Int Std. #: SWL1000
Head Pressure: 8 psi	Mass Defect: 0	Step #4	315	0.0	16.4	38	Tune Std. #: WL-367
	Background Mass: 35 amu	Inject.	45	0.0	0.5	0.5	Inj. Vol.: 1 ul.
	Curve Date: 10/1 MK	Inject.	300	100	2.55	3.5	DFTPP Area 198: 715361
		Inject.	300	0.0	15	15.05	DDT Breakdown: none

Sample / File ID	Client	Inj Time	Dil	Matrix	Fraction	Method	Notes
DFT12116		16:46	SV		DFTPP	P270	DFTPP passed
10C1211A		16:53	SV		BNA	P270	cond. cal std., CCCA, SPCCs pass
10A91211		17:53	SV		Acids	P270	
10P1211A		18:46	SV		Acids/BNA	P270	
MPB1076	EVS	19:53	SV	WV	Acids/BNA Pest	P270	BNA 12/11/98 EVS
UPB1076W		20:59	SV	WV	BNA	P270	
212644		21:12			Acids/BNA Pest	P270	
212645		22:02					
212646		22:52					
212647		23:37					
212676MS		00:40			BNA	P270	

133_RUN.XLS

Approved By: [signature] 12/14/98
QA Manager

Approved By: [signature] 12/14/98
Area Manager

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

SEMI-VOLATILE LABORATORY MASS SPECTROMETRY CORRECTIVE ACTIONS

APPROVALS:

Organic Manager: _____ Date: 2/3/98
Jeff P. Glaser

QA Manager: _____ Date: 2/3/98
Rick D. Wilburn

Laboratory Manager: _____ Date: 2/3/98
Douglas E. Kriscunas

Procedure Number: GR-04-101
Revision Number: 3.0

Date Initiated: 12/19/96
Effective Date: 3/2295

Date Revised: 2/3/98
Pages Revised: All

By: Jeff P. Glaser

Total Number of Pages: 11

1.0 SUMMARY OF PROCEDURE

1.1 Sample results must conform to specified method quality control criteria. Every effort will be made to produce results that conform with those criteria. When this is not possible and an out of control data point occurs, it is important for the analyst to recognize the problem, then qualify and narrate the data correctly.

1.2 This SOP provides guidelines for corrective actions regarding out of control sample data points. It provides instructions on both the procedure to follow when a data point is out of control, and how to narrate and qualify the data when necessary. It is the responsibility of the analyst to inform the project chemist when a situation arises where he feels it is important to notify the client now, rather than when they receive the data package. The analyst must to do everything in his power to prevent the qualification of data when possible. Re-injections, re-extractions, and alternate cleanup methods will all be employed when practical to do so.

1.3 Non-sample related quality control deviations such as out of control tunes or continuing calibrations are not covered in this SOP. Those items are covered in the appropriate analytical SOP. The more common instances where a sample will require qualification have been covered in this SOP. Analyst experience will govern the correct course of action in other situations.

2.0 DETAILED PROCEDURE

2.1 Surrogate Failure

2.1.1 When a surrogate fails recovery criteria, deciding on how to proceed is influenced by a number of factors. Is re-injection required or should the sample be re-extracted? Does the data require qualification, and if so how should the data be qualified? Analyst experience is critical in determining the correct course of action. Surrogate failures generally fall into three categories: sample matrix interference, extraction error, or instrument failure. The course of action taken is specific to those three categories, and by the following general rules. See also Attachment 1.

a) If any surrogate has a percent recovery <10%, all data and detection limits for the corresponding fraction will be narrated and qualified as unusable. (See LIMS qualifier # 17 and 18).

b) If any surrogate has a percent recovery of ≥10%, but the percent recovery is less than the laboratory generated recovery window, all positive hits and detection limits will be narrated and qualified as estimated for that fraction. (See LIMS qualifier # 16).

c) If any surrogate has a percent recovery that exceeds the laboratory generated recovery window, all positive hits for that fraction will be qualified and narrated

as being estimated. No qualification is required on detection limits. (See LIMS Qualifier # 15).

d) For all non-diluted runs, or runs with dilutions of ≤ 1:5, surrogate recoveries will be calculated and reported, and data will be narrated and qualified when necessary. Surrogate recoveries will not be calculated, reported, or used to qualify data on samples run at a dilution of >1:5. (See LIMS qualifier # 20).

e) Surrogate recoveries outside of the laboratory generated windows will not be used to qualify quality control samples with acceptable spike compound recoveries. However, if a matrix spike or matrix spike duplicate has acceptable surrogate recoveries, and the non-spiked version of the sample has surrogate recoveries outside of the acceptable windows, the data will be narrated and qualified, or appropriate action will be taken according to rules outlined elsewhere in this section.

2.1.1.1 Sample Matrix

2.1.1.1.1 Surrogate failure due to sample matrix problems will be suspected when one or more of the following occur:

a) poor chromatography
b) raised baseline
c) only one or two surrogates are out of control
d) co-elution of the surrogate with another peak

If sample matrix <u>is</u> suspected to be the cause of a failed surrogate recovery and no dilution is required, the sample will not be re-run and positive results will be narrated and qualified as estimated. All results less then the detection limit will be considered as unusable. (See LIMS qualifier # 19). If a dilution of ≤1:5 is required, the surrogates from the diluted run will be used to decide if qualifications are necessary.

2.1.1.1.2 If sample matrix <u>is not</u> suspected to be the cause of a failed surrogate recovery, and a dilution of >1:5 is necessary, surrogate results will not be reported and no data will be qualified. (See LIMS qualifier # 20). If the sample was diluted ≤1:5, the surrogates from the diluted run only will be used and reported.

2.1.1.1.3 If a sample was run at a dilution of >1:5 due to matrix interference, surrogate recoveries will not be reported, and no qualifications re necessary. (See LIMS qualifier # 53).

2.1.1.2 Extraction Error

2.1.1.2.1 If surrogates are out of the laboratory generated recovery windows, extraction error will be suspected if:

a) the analysis of extracted samples run before and after the sample in question had acceptable surrogate recoveries
b) the internal standard areas are acceptable/mis-injection is not assumed
c) base line sensitivity appears normal
d) most or all of the surrogates are out of control and there are no apparent matrix related problems

2.1.1.2.2 If the sample is within hold time it will automatically be re-extracted. If the re-extraction works the first run will not be used and no data will be qualified. If the re-extraction shows similar results, matrix interferences will be assumed and the data for that fraction will be narrated and qualified as estimated (see Section 2.1.1.1). The initial run will be the run that is reported. If the sample is out of its' hold time, the project chemist will be consulted and the analyst will proceed as instructed, by either qualifying the data for that fraction as estimated based on poor surrogate recoveries, or by having the sample re-extracted. If the re-extract works, the data will be narrated and qualified because it was re-extracted outside of the hold time. (See LIMS qualifier # 1).

2.1.1.3 Instrument Failure

2.1.1.3.1 If surrogates are out of the laboratory generated recovery windows mis-injection or instrument failure will be suspected if:

a) other runs produced inconsistent results
b) the internal standard areas are not acceptable
c) base line sensitivity looks low
d) previously injected samples had poor matrixes which may have affected surrogate recoveries of the sample in question

2.1.1.3.2 If instrument failure is assumed the sample will automatically be re-run. If the re-run works the first run will not be used and no data will be qualified. If the re-run does not work matrix interferences will be assumed and the analyst will proceed according to Section 2.1.1.1 or 2.1.1.2 as appropriate.

2.1.2 Surrogates will also fail when the sample has been mis-spiked with internal standard. Corrective action for this is outlined in section 2.2.

2.1.3 If one or more of the surrogates are slightly out of control and none of the above situations appear to apply, the sample will be re-injected. If surrogates are still out of control, and there is no reason to assume the instrument is out of control, matrix interferences or extraction error will be assumed. Proceed as directed in 2.1.1.2.2. Reference also Attachment 1, the flowchart provided.

2.2 Internal Standard Failure

2.2.1 Any time an internal standard fails the -50% +100% area criteria, the ability to accurately quantitate an analyte is lost. For that reason every effort will be made to prevent the failure of an internal standard. Internal standards can fail their area count criteria for the following reasons: software failure, matrix interferences, mis-spiked, or instrument failure.

2.2.1.1 Software Failure

2.2.1.1.1 Samples with poor matrixes or some sort of chromatographic interferences may cause the quantitation software to mis-integrate the peak. If an internal standard fails its area criteria under these circumstances, the data will be reviewed for mis-integration. If this solves the problem, no narration or qualification will be necessary, if not, proceed as directed below.

2.2.1.2 Matrix Interferences

2.2.1.2.1 If, even after manual integration, a matrix interference is preventing accurate internal standard area quantitation, the sample will be either:

a) re-ran, or
b) diluted and re-spiked with internal standard to bring the internal standard concentration back up to its original extract concentration.

The correct course of action is dependent on the extent of the matrix interferences. Since specific compounds go with specific internal standards, if the sample is diluted and re-spiked both the non-diluted and diluted runs will be combined and reported as one. The analysis date reported will be the date the initial run was made. The elevated detection limits reported for the compounds associated with the diluted run will be explained in a narration stating why the dilution was made. (See LIMS qualifier # 7). As long as the internal standard area is acceptable in the diluted run, no data qualifications will be made based on internal standard areas. Exceptions to the rule will exist when the non-diluted run has matrix problems outlined elsewhere in this section. In these instances only the diluted run will be used.

2.2.1.3 Mis-Spiked

2.2.1.3.1 If surrogate recoveries and internal standard areas fail their respective criteria, the data will be reviewed for a possible mis-spike of the internal standard. If upon review the analyst determines that the sample was mis-spiked with internal standard, the sample will be re-quantitated according to the concentration of the internal standard present in the extract. If the level of internal standard present in the extract cannot be determined, and the samples hold time has not expired, the sample will be re-extracted, re-spiked and re-run. No data will be qualified as long as the re-extract works. If the sample is out of its' hold time, the project chemist will be consulted and the analyst will proceed as instructed, by either qualifying the data for that fraction as estimated based on poor internal standard recoveries, or by having the sample re-extracted. If the re-extract works, the data will be narrated and qualified because it was re-extracted outside of the hold time. (See LIMS qualifier # 1).

2.2.1.4 Instrument Failure

2.2.1.4.1 If the internal standards fail their criteria, but the surrogates pass, the sample was probably mis-injected. The sample will be re-run. No narration or qualification will be necessary if the re-run works.

2.3 Dilution Procedures

2.3.1 Samples will be diluted when there is:

a) sufficient cause to believe that the ability to see the non-diluted detection limits is in question.
b) a chromatography problem from a matrix interference which may be interfering with the recovery of target analytes.
c) sufficient cause to believe that the matrix of the sample will be detrimental to the instrument.

2.3.2 Elevated detection limits will never be reported based on the belief that the non-diluted detection limits cannot be achieved. The sample will always be diluted and the elevated detection limits reported from the results of the diluted run.

2.3.3 The minimum dilution possible will be made that allows the analyte with the highest concentration to be kept near the upper half of the linear range of the instrument. If the dilution is being made based on matrix problems rather than elevated target analyte concentrations, the minimum dilution possible will be made. The sample will be re-run at a lesser dilution if initially over diluted to accomplish this.

2.3.4 Under normal operating conditions, if a sample was initially run straight and a dilution is required, all results and detection limits will be reported from the diluted run only (except as noted above in 2.2.1.2.1).

2.3.5 Diluted samples will be narrated explaining the reason for the elevated detection limits unless it is obvious due to the high target concentrations being reported. (See LIMS qualifier # 7).

2.4 Laboratory Fortified Blank (LFB) Failure

2.4.1 Failure of any part of an LFB requires qualification or when possible, re-extraction of the samples in that batch looking for the failed compound. If any compound or compounds in the Laboratory Fortified Blank (LFB) exceed the recovery criteria, positive results for that analyte for every sample in the extraction batch will be estimated. All results less than the detection limit are acceptable and need no qualification. (See LIMS qualifier # 66). However, if any compound or compounds in the LFB are out of control low, positive results for that analyte for every sample in the batch will be qualified as estimated and the detection limit for that analyte will be considered approximate. A batch narrative will be written any time a LFB analyte fails recovery criteria. (See LIMS qualifier # 5).

2.5 Matrix Spike (SPK1 and SPK2)/Matrix Spike Duplicate (MSD) Failure

Approved By: _____ 2/3/98
QA Manager

Approved By: _____ 2/3/98
Area Manager

2.5.1 If all analyte recoveries are within laboratory generated windows on both SPK1 and SPK2, but the precision for the MSD is out, the data for the failed compound in the non-spiked version of the sample will be qualified as estimated. (See LIMS qualifier # 3).

2.5.2 If the percent recovery for an analyte is in control in one SPK, out of control in the other SPK, and the MSD percent difference is in control, no data will be qualified, but a narrative will be written. (See LIMS qualifier # 63).

2.5.3 If any single analyte recovery is outside the laboratory generated windows on either or both SPK1 and SPK2, and the MSD precision is out, the data for the failed compound in the non-spiked version of the sample will be qualified as estimated. (See LIMS qualifier # 2).

2.5.4 If any spiked compound fails in the either or both SPK1 and SPK2, and in the LFB, the data for that analyte for every sample in the batch will be estimated. (See LIMS qualifier # 5).

2.5.5 If SPK1 and/or SPK2 percent recoveries for any analyte are above the upper control, and the results for that analyte in the sample are less than the reporting limit, then no qualification is required. (See LIMS qualifier # 68).

2.5.6 If a sample has a concentration of a spiked analyte that is greater than five times the spiked concentration, and the resulting spike recoveries for SPK1 and/or SPK2 and outside of the control limits, no qualification to sample results is necessary. (See LIMS qualifier # 86).

2.5.7 If the matrix spike recoveries are unable to be obtained due to high background matrix interferences, high analyte concentration, or interfering peaks, then the sample will be qualified for each analyte affected. (See LIMS qualifier # 23, 24, and 25 respectively.)

2.6 Extracted Method (MPB) or Daily Instrument Blank (BLK) Failure

2.6.1 The MPB or BLK will fail for two reasons:

a) surrogate recoveries in the MPB are outside of the laboratory generated windows

b) either the MPB or the BLK are contaminated with one or more target analytes

2.6.2 If the surrogate recoveries in the MPB are above the laboratory generated windows, data will be narrated but not qualified. (See LIMS qualifier # 15). If surrogate recoveries are below the laboratory generated windows, the MPB must be viewed as mis-extracted and cannot be used to determine sample contamination from the extraction process. Any consistent positive hits from all the samples extracted that day must be narrated and qualified as estimated. (See LIMS qualifier # 17 and 18).

2.6.3 If the BLK is contaminated with any target analytes, analysis may not begin. The problem must be corrected before sample analysis can re-commence.

2.6.4 Sample data will never be blank subtracted. Positive hits found in the MPB and the sample will be reported, and narrated in an analysis narrative. The data for that compound will be reported as estimated. (See LIMS qualifier # 21).

2.6.5 A BLK will not be run when a clean MPB is run. In this case the MPB is also the BLK and will be reported both ways.

2.7 Missed Holding Times

2.7.1 All samples that are analyzed beyond their established USEPA maximum holding times must be qualified. All positive results must be qualified as estimated, and all non-detectable results must be qualified as approximate. (See LIMS qualifier # 1). If the hold time is missed due to laboratory error, a detailed explanation must be provided with the qualifier.

2.7.2 All samples that are received by the laboratory that have already exceeded their USEPA maximum allowable holding time must have their results qualified as estimated. (See LIMS qualifier # 12).

3.0 REPORTING AND DELIVERABLES

3.1 All out of control data must be narrated or qualified as appropriate, using the conventions required by the laboratories LIMS system. It is extremely important that the end user of the data be informed of all data which requires some sort of qualification or narration.

4.0 QUALITY ASSURANCE

4.1 Quality assurance procedures are covered in section 2.0.

5.0 REFERENCES

5.1 For further information, consult the Method 8270 SOP, or the TriMatrix Laboratories, Inc. Quality Assurance Manual.

5.2 USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review. Office of Emergency and Remedial Response, U.S. Environmental Protection Agency, Washington, DC 20460. Publication 9240.1-05, PB94-963501, EPA540/R-94/012, February, 1994.

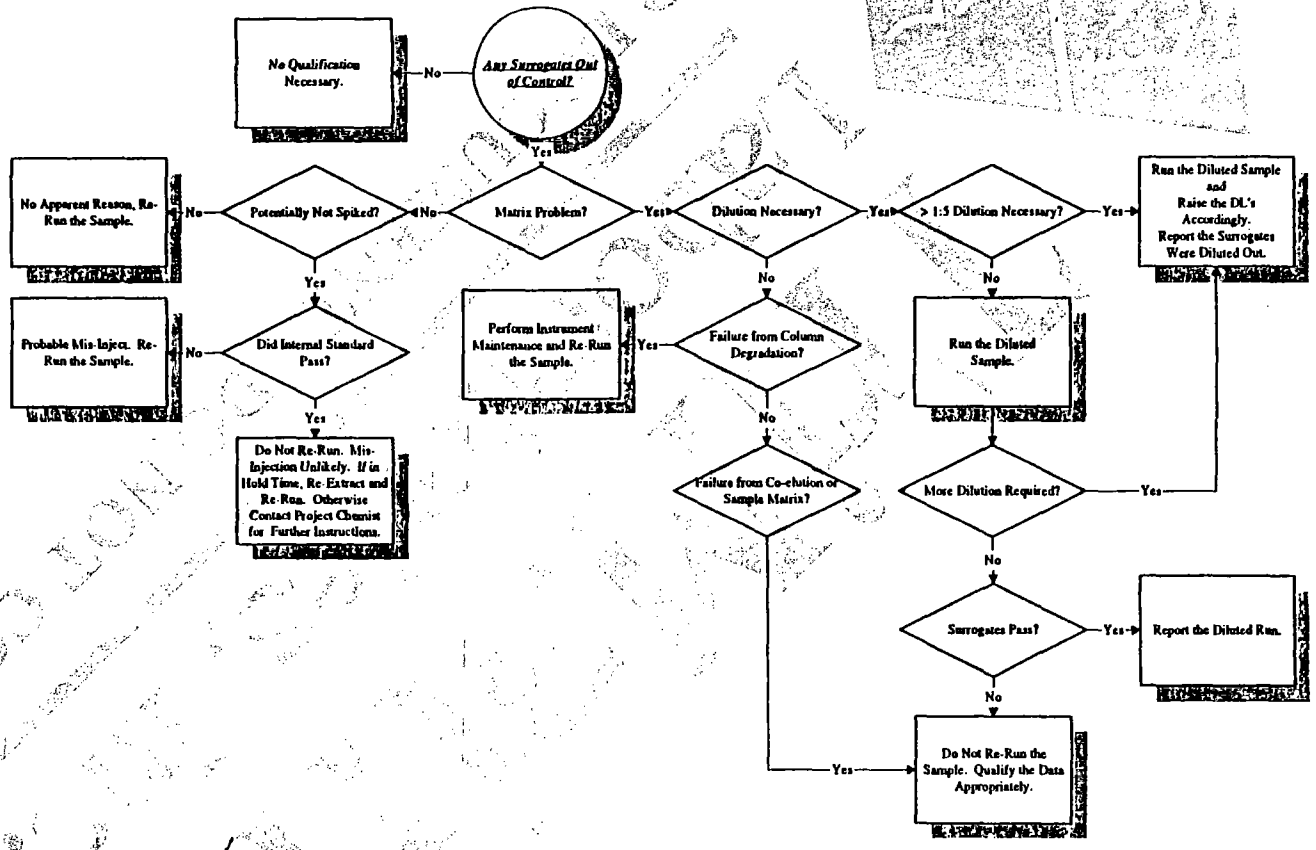
6.0 ATTACHMENTS/APPENDICES

6.1 Attachment 1: Flowchart for the Determination of Sample Re-Run Due to Surrogate Failure

Approved By: _____ 2/3/98 QA Manager

Approved By: _____ 2/3/98 Area Manager

Attachment 1
Flowchart for the Determination of Sample Re-Run Due to Surrogate Failure



Approved By: _____ 2/3/98
QA Manager

Approved By: _____ 2/3/98
Area Manager

Procedure No. 1
Low Flow Purging and Sampling

LOW FLOW PURGING AND SAMPLING SOP

PURGING PROCEDURES

- Measure depth-to-Water and Depth-to-Bottom of each well

- Calculate one volume of the screened or open intervals as performed in the following example (if using packer):

  10 foot screen, 2-inch well diameter = 10 feet x 0.1632 gallons/foot = 1.632 gallons/volume
  1.63 gallons x 3.785 liters/gallon = 6.17 liters or 6.17 liters x 1,000 mls/liter = 6171 mls
  Assuming 200 mls/min purge rate: 6171 mls ÷ 200 mls/min = 30.85 minutes to purge one volume

  Calculating the purge volume will be helpful in determining when stability of the water-bourne constituents can be expected.

- Lower pump to the mid-point of the screened interval.

- Inflate packer (if used) just above top of screen to isolate the screened interval.

- Begin to purge well. USEPA recommends a purge rate of 200 to 300 mls/min. Actual purge rates will be site specific. If the packer is not used, the purge rate should not exceed the recharge rate (i.e., no drawdown in static water level).

- Measure pH, Temperature, Conductivity, Redox Potential and Dissolved Oxygen with a flow-through cell at 3 to 5 minute intervals. Also measure Turbidity, at 3 to 5 minute intervals. These parameters should begin to stabilize before one-half the screened area volume is removed.

- Once the following conditions occur over 3 consecutive readings, sampling can begin:

  - pH stabilizes to within 0.1 units

  - Temp, Redox, D.O. and Turbidity have stabilized to within 10%

  - Conductivity has stabilized to within 10%

  Note: In Region III, Conductivity should be within 3%.

SAMPLING PROCEDURES

- Using the well purging pump already in place, reduce flow rate to 100mls/min for VOCs and sample out of the pump discharge line. The sampling rate can be increased to 200-300 mls/min for the remaining parameters. The flow-through cell should be disconnected or bypassed during sample collection.

- Fill sample containers for appropriate analyses in the following order:

  1. Volatile organics and total organic halogens
  2. Dissolved gases and total organic carbon
  3. Semivolatiles
  4. Metals and cyanide
  5. Major ions
  6. Radionuclides

- At completion of sampling, the packer will be deflated (if used) and the pump removed.

DECONTAMINATION OF EQUIPMENT

- The pump will be disassembled and components (including the packer, if used) will be decontaminated in the following manner for sites with organic contaminants:

  - Alconox and water wash;
  - Tap water rinse;
  - Methanol (or acetone) rinse;
  - Triple distilled water rinse; and
  - Air dry and store pump in plastic

The following decontamination procedures will be followed when working at sites with inorganic contaminants:

- Alconox and water wash;
- Tap water rinse;
- Hydrochloric Acid rinse (0.1N);
- Triple distilled water rinse; and
- Air dry and store pump in plastic

When working at sites with both organic and inorganic contaminants, the first procedure (for organic contaminants) should be followed with the HCl rinse followed by a distilled water rinse added after the tap water rinse.

After decontamination of the pump and components (including the packer, if used), two volumes of distilled water will be flushed through the pump assembly to ensure all decontamination fluids have been removed. This volume is approximately 0.75 gallons. It is beneficial to pump the distilled water at high speed to effectively remove decon. solutions.

This SOP was developed based on articles by the USEPA and various experts in the field including Barcelona, Puls, Korte, and Kearl. This SOP may be edited periodically based on new research and regulatory requirements.

REFERENCES

USEPA (November 1992) RCRA Ground-water Monitoring: Draft Technical Guidance. Office of Solid Waste EPA/530-R-93-001

Kearl, P.M., Korte, N.E., and T.A. Cronk (1992) Suggested Modifications to Ground Water Sampling Procedures Based on Observations from the Colloidal Borescope

Puls, R.W., and M.J. Barcelona (1995) Low-Flow (minimal drawdown) Ground-water Sampling Procedures. USEPA Office of Research and Development EPA/540/s-95/504

PROCEDURE NO. 5

ELECTRONIC WATER LEVEL INDICATOR

PROCEDURE NO. 5

ELECTRONIC WATER LEVEL INDICATOR

1.0 INTRODUCTION

This document describes the general procedures for acquiring static water levels in wells. The electronic water level indicator gives an audible signal or meter reading when the probe at the end of the tape completes an electric circuit by contacting water. The tape is divided into measured increments. Water level measurements should be taken to the nearest .01 foot.

2.0 PROCEDURES

2.1 PREPARATION

Prior to beginning a field investigation, the staff personnel who will be operating the equipment will complete the following tasks:

1. Coordinate schedules/actions with supervisor.

2. Schedule equipment use with FEM.

3. Pick-up equipment on scheduled date and inspect equipment with FEM.

4. Ensure the proper operation of equipment prior to leaving for the site (this includes having fully charged batteries).

2.2 OPERATION

The following procedures will be used to measure static water levels with an electronic tape:

1. Check the probe and tape for visible contamination and decontaminate both between each measurement location following Procedure 6.

2. Lower the probe into the well by pulling the tape from the hand held reel. When raising and lowering the tape, take care not to rub it against the top of the well casing, which can strip insulation off of the wires.

3. As the tape is lowered, straighten any kinks or bends in the tape which may alter the length the tape.

4. Continue lowering the tape until the audible signal is heard.

5. Raise and lower the tape a short distance to confirm the point at which water is contacted.

6. Measure and record the length of tape in the well to the nearest .01-foot; measure from the established monitoring point at the top of the well casing.

7. Record the well number or location, date and time of measurement, field inspector initials, water level measurement, total depth of the well, and any problems, unusual measurements, or damage to the well in the field logbook.

If the electronic water level indicator does not appear to work, check the following items:

- Turn on the probe and press the test button to confirm that the unit is working.

- If the test button does not activate the tone, turn on the probe and dip the tip in water to see if the test button is malfunctioning.

- Check and replace the battery if necessary which is located inside the reel.

- If the probe still does not work, return the unit to the manufacturer.

If the electronic water level indicator works intermittently, or the probe feels like it has contacted water but provides no indication, check the following items:

- Carefully check the condition of the plastic-coated wires (tape) which attach to the probe. If the insulation on these wires appears to be damaged, return the probe to the office. Make a note of this condition on any records of water level measurements. Damaged insulation may produce erroneous water level measurements.

- Check the probe for the presence of free-phase hydrocarbons, such as oil or gasoline, which may float on top of the water. These hydrocarbons are non-conductive, and will not activate the electric probe. Make a note of the presence of free-phase hydrocarbons on any records of water level measurements.

2.3 DATA REDUCTION AND INTERPRETATION

Water level measurements will be subtracted from the elevation of the established monitoring point on the top of the well casing. This will provide the actual static water level in the well in terms of the established elevation datum (preferably mean sea level). If measurements are obtained in the same water zone, the hydraulic gradient and flow direction of the water zone can be determined.

2.4 POST OPERATION

2.4.1 Field

If the equipment has come in contact with contaminated materials, decontaminate all equipment as described in Procedure 6. Always decontaminate the probe and tape after every use.

Prior to leaving the site each day the following items will be completed:

1. Record all observations and notes in the logbook.

2. Complete all logbook entries which will include: project name and number, date, field time, personnel, visitors on-site, weather conditions, site conditions and any other pertinent data.

3. Review logbook entries and verify accuracy of entries. Sign the last page of the days' entry.

2.4.2 Office

When returning the instrument and supplies to the FEM, the following items will be completed.

1. Report any equipment malfunction and/or damage.

2. Copy field logbook and any field maps for project file.

PROCEDURE NO. 6

EQUIPMENT DECONTAMINATION

PROCEDURE NO. 6

EQUIPMENT DECONTAMINATION

1.0 INTRODUCTION

Decontamination procedures are performed to ensure that no contaminants are introduced into samples, spread across the site, or carried between sites. In general, equipment decontamination should be performed on-site at the start of the site activities, between sampling events, and at the completion of the site activities. A known contaminated site location will be sampled last to reduce the chance of cross contamination with "clean" areas. The following sections contain a general discussion of various decontamination procedures.

2.0 PROCEDURES

2.1 PREPARATION

Prior to beginning a field investigation, the staff personnel who will be decontaminating the sampling equipment will complete the following tasks:

1. Coordinate schedules/actions with supervisor.

2. Schedule equipment use with the Field Equipment Manager (FEM).

3. Pick up equipment on scheduled date and inspect equipment with FEM. (This includes having distilled water and standard decontamination solutions.)

4. Ensure that all of the decontamination equipment is assembled prior to leaving for the site. At a minimum this includes the following items:

- Wash pails or tubs (plastic or metal);
- Scrub brushes;
- Paper towels;
- Acetone (pesticide grade) or other appropriate solution
- Latex gloves;
- Plastic trash bags;
- Zip-lock bags;
- Nitric acid solution (if sampling for metals analyses)
- Aluminum foil;
- Plastic sheeting;
- Non-phosphate detergent (such as Alconox);
- Distilled/deionized water; and
- Decon water containers.

2.1.1 Potable Water

A potable water supply is available on the site.

2.1.2 Decontamination Solutions

Decontamination solutions will be collected for QA/QC purposes. The field decontamination solutions will be collected in the appropriate sample jars provided by the laboratory.

2.1.3 Decontamination Station Set Up

A permanent decontamination facility exists on-site. This facility consists of a crushed stone wash pad draining to a collection sump. High pressure water spray decontaminates large pieces of equipment or vehicles.

Smaller equipment items such as bailers, trowels and containers will be decontaminated by establishing a small decontamination station adjacent to the work area. The station will contain the previously listed items.

2.2 STANDARD DECONTAMINATION PROCEDURES

Equipment will be decontaminated using the following sequence:

- Non-phosphate detergent (Alconox) plus tap water wash.
- Tap water rinse
- Distilled/deionized water rinse
- 10 percent nitric acid rinse*
- Distilled/deionized water rinse*
- Acetone (pesticide grade) rinse**
- Total air dry or pure nitrogen blow out**
- Distilled/deionized water rinse**

* Only if sample is to be analyzed for metals.

** Only if sample is to be analyzed for organics.

Whenever possible samplers should be numbered in a manner that will not affect their integrity and wrapped in a material (e.g., aluminum foil) that has either been autoclaved or cleaned in the same manner as the sampler. Equipment should be custody sealed and information concerning decontamination methodology, date, time, and personnel should be recorded in the field log book.

The use of distilled/deionized water commonly available from commercial vendors may be acceptable for sampling equipment decontamination provided that it has been verified by laboratory analysis that the water has been distilled and deionized.

2.3 DISPOSAL OF DECONTAMINATION SOLUTIONS AND SUPPLIES

Disposable equipment and supplies will be left at the work site in appropriate containers. Proper disposal of the waste will be arranged by Gould's representatives.

Decontamination solutions can usually be placed on the landfill surface as dust control, away from any test wells or areas where future soil tests might be collected.

PROCEDURE NO. 7

pH METER

PROCEDURE NO. 7

pH METER

1.0 INTRODUCTION

This document describes the general procedures for acquiring the pH and temperature of solutions.

The following quality assurance procedures apply to all water quality instruments used during data acquisition.

1. All water quality instruments will be operated according to operating instructions supplied by the manufacturer. A copy of the operating manual will be kept with the instrument.

2. Battery voltage levels for all instruments will be monitored each day. Batteries will be charged or replaced when voltage levels fall below the level specified by water quality equipment manufacturers.

3. All water quality instruments will be calibrated monthly by the field equipment manager (FEM). Calibration records will be maintained in the office where the instrument is stored. More frequent calibrations may be necessary if field measurements indicate possible instrument malfunction.

2.0 PROCEDURES

2.1 PREPARATION

Prior to beginning a field investigation, the staff personnel who will be operating the equipment will complete the following tasks:

1. Coordinate schedules/actions with supervisor.

2. Schedule equipment use with FEM.

3. Pick-up equipment on scheduled date and inspect equipment with FEM. (This includes having distilled water and standard solutions to calibrate the meter.)

4. Ensure the proper operation of equipment prior to leaving for the site (this includes having fully charged batteries).

2.2 OPERATION

2.2.1 Calibration

The following two-buffer calibration will be done prior to each day in which the instrument is used in the field.

1. Slide power switch on meter to the on position.

2. Slide mode switch to pH. If LO BATT indicator on LCD remains on, battery must be replaced.

3. Attach shorting plug (BNC connector) to connector on top of meter. Slide mode switch to pH. Adjust CALIB. knob to read a 7.00.

4. Remove shorting plug and attach pH electrode probe to connector.

5. Plug temperature probes (ATC) into the two input jacks to the left of the electrode probe on meter.

6. Select two buffers.

7. Place the pH electrode probe and temperature probe in one of the buffer solutions to a depth of two cm and stir moderately. Allow reading to stabilize and set temperature control (°C/slope) to the temperature of the solution. Adjust calibration until the display indicates the pH of the buffer at the solution temperature.

8. Remove both probes from the first buffer solution and rinse by stirring moderately in distilled water. Shake off excess drops of water.

9. Place both probes and ATC probe in the second buffer solution to a depth of about two cm and stir moderately. Allow reading to stabilize and adjust the slope control until the pH of solution temperature is displayed.

10. Rinse both probes with distilled water.

11. Place both probes in first buffer solution to see if meter is holding its calibration. Allow reading to stabilize and if reading is greater than 1%, recalibrate.

12. Rinse both probes in distilled water.

2.2.2 Data Collection

Samples will be collected in the field in clean unused sample jars and tested immediately. The following procedures will be followed:

1. Place electrode probe and ATC probe in unknown sample and allow reading to stabilize. Set temperature control to temperature of sample. Read the pH of sample and pick a buffer solution nearest to the reading.

2. Remove probes from sample and rinse by stirring moderately in distilled water.

3. Place probes into selected buffer and allow reading to stabilize. Set temperature control to temperature of buffer. Adjust CALIB knob on meter if necessary to equal pH of buffer.

4. Remove probes and rinse by stirring moderately in distilled water.

5. Re-measure the unknown solution by placing probes in the solution and allowing the reading to stabilize.

6. Record location, date, time, operator, and results in the field logbook.

2.2.3 Data Reduction and Interpretation

There is no data reduction required for this instrument. Because the instrument measures indicator parameters, interpretation of data is straightforward.

2.3 POST OPERATION

2.3.1 Field

If the equipment has come in contact with contaminated soils or materials, decontaminate all equipment as described in Procedure 6. If the equipment has become dirty, be sure to clean off visible dust or dirt. Always clean probes with distilled water and wrap with damp paper towel immediately after every use. Damage can occur to the electrode if an acidic, alkaline, or organic rich solution is left on the electrode.

Prior to leaving the site each day the following items will be completed:

1. Record all observations and notes in the logbook.

2. Complete all logbook entries which will include project name and number, date, field time, personnel, visitors on-site, weather conditions, site conditions and any other pertinent data.

3. Review logbook entries and verify accuracy of entries. Sign the last page of that day's entry.

2.3.2 Office

All water quality instruments will be cleaned in the laboratory by the FEM before the instrument is used again. When returning the instrument and supplies to the FEM, the following items will be completed:

1. Report any equipment malfunction and/or damage.

2. Copy field logbook for project file.

3. Copy any field maps used for project file.

REFERENCE SOURCES

Operating Manual for pH meter, Orion

Orion Research Incorporated
Laboratory Products Group
529 Main Street, Boston, MA 02129
Telephone (617) 242-3900

PROCEDURE NO. 8

SPECIFIC CONDUCTIVITY METER

PROCEDURE NO. 8

SPECIFIC CONDUCTIVITY METER

1.0 INTRODUCTION

This document describes the general procedures for measuring the specific conductivity of solutions. The meter displays conductivity in five ranges from 0 to 20,000 micromhos/centimeter (umhos/cm) and has an internal thermistor for automatic temperature compensation.

The following quality assurance procedures apply to all water quality instruments used during data acquisition.

1. All water quality instruments will be operated according to operating instructions supplied by the manufacturer. A copy of the operating manual will be kept with the instrument.

2. Battery voltage levels for all instruments will be monitored each day. Batteries will be charged or replaced when voltage levels fall below the level specified by water quality equipment manufacturers.

3. All water quality instruments will be calibrated monthly by the field equipment manager (FEM). Calibration records will be maintained in the office where the instrument is stored. More frequent calibrations may be necessary if field measurements indicate possible instrument malfunction.

2.0 PROCEDURES

2.1 PREPARATION

Prior to beginning a field investigation, the staff personnel who will be operating the equipment will complete the following tasks:

1. Coordinate schedules/actions with supervisor.

2. Obtain appropriate permission for site access.

3. Schedule equipment use with FEM.

4. Pick-up equipment on scheduled date and inspect equipment with FEM. (This includes having distilled water and standard solutions to calibrate the meter.)

PROCEDURE NO. 19

SOIL SAMPLING

PROCEDURE NO. 19

SOIL SAMPLING

1.0 INTRODUCTION

There are a wide variety of sampling tools and methods for obtaining soil samples. The proper selection of soil sampling tools and methods will be dependent on a number of factors including soil gradation and density, sampling depths, and contaminants of concern. Soil samples will be collected with hand tools and drilling equipment.

2.0 SHALLOW SOIL SAMPLING

Shallow soil samples will be obtained with shovels, trowels and hand augers. Hand augers will be used to perform shallow borings and collect soil samples for chemical analysis. These soil samples are typically quite disturbed, but are useful for soil classification and to identify the extent of shallow contamination. The effective depth for hand auger borings will vary greatly depending on site conditions, but is typically not deeper than five feet. Hand auger borings are not appropriate for sites with cobbles, hard soils, frozen ground, debris, or soils which tend to cave in.

2.1 EQUIPMENT

The bucket-type hand auger consists of a stainless steel bucket with two cutting edges at the tip. The dutch hand auger is used in clayey soils, and consists of an open double helix arrangement.

The following items will be available when conducting shallow soil sampling:

- Hand auger(s);
- Rods, handle, and pipe wrenches (for hand auger);
- Shovel, pick, and trowel;
- Sample jars and labels;
- Cooler and ice;
- Decontamination supplies and distilled/deionized water;
- Plastic sheeting;
- Containers for mixing composite samples;
- Waterproof magic marker;
- Flagging tape;
- Wooden stakes;
- Folding ruler and measuring tape (50 or 100-foot); and
- HNu or other instrumentation required by the site specific sampling plan.

General Procedures

The following procedures are generally used when sampling with hand augers:

1. The hand auger boring location will be determined from a site plan;

2. Vegetation or loose surface debris which could fall in the hole during augering will be cleared away;

3. One-foot intervals will be marked on the auger rod with a waterproof marker;

4. The hand auger will be decontaminated as appropriate and rinsed in distilled/deionized water;

5. Each interval of soil which is removed will be visually examined for changes in soil type, and will be scanned (e.g. HNu) for any field parameters which may be required by the sampling plan;

6. All observations and depths will be recorded on the hand auger boring log;

7. The marks on the hand auger rod will be used to measure the depths at which samples are collected;

8. Soil samples will be collected at required intervals, or based upon field observations;

9. At the completion of each boring, the hole will be backfilled with soil, or grout if required by a site specific plan;

10. The location of the boring will be measured relative to known site features, or recorded locations on a site plan; and

11. Location and elevations will be surveyed at a later date, the boring locations will be marked with wooden stakes and flagging tape. The boring number will be written on each stake with a waterproof marker.

Sample Collection

Soil samples are generally removed through the top of the bucket auger. Depending on the soil consistency, the auger may be gently tipped upside down, or the auger handle hit on the ground to extract the sample.

Samples collected with a hand auger will often contain debris or soil which may have fallen into the top of the hand auger bucket during the augering process. As the sample is removed from the auger bucket, the upper portion of the sample will be discarded, and not submitted for analysis.

For "grab" samples, the sample will be transferred directly from the auger bucket to the sample jar. For "composite" samples, the auger spoils will be placed into a container and thoroughly homogenized before placing in the sample jar.

Appropriate marking of the sample jars, sample documentation and handling of the samples will be performed as outlined in the respective procedures documents.

All equipment will be thoroughly decontaminated between each boring, and before leaving the site as outlined in the equipment decontamination procedures.

3.0 DEEP SOIL SAMPLING

Deep soil samples are commonly obtained from test pits or through the use of drilling equipment and downhole samplers. The most common type of downhole sampling device is the split spoon sampler.

A split spoon sampler commonly is used for the collection of soil samples at depths greater than five feet. A drill rig will be employed to advance the borehole to the desired sampling depth, using either rotary or hollow stem auger drilling techniques. The sampling plan will reference the specific sampling depths, the frequency of sampling, and volume of soil that is needed. Once the desired depth is reached a decontaminated split spoon sampler will be advanced a specific vertical distance by means of a 140-pound free falling weight (ASTM D 1586).

The split spoon sampler consists of steel tubing split longitudinally and equipped with a drive shoe and head. The sampler is available in a variety of lengths and diameters. Procedure No. 11 discusses the methods used to obtain a split spoon sample.

PROCEDURE NO. 29

SEDIMENT SAMPLING

PROCEDURE NO. 29

SEDIMENT SAMPLING

1.0 INTRODUCTION

The following section outlines the recommended procedures and equipment for the collection of representative sediment samples for chemical analysis from standing lakes, ponds, lagoons, and impoundments, and flowing streams, rivers and channels.

The collection of the sample is highly dependent on site specific conditions. The following factors will be considered prior to collection:

1. Access to the sample location;

2. Flow rate; and

3. Seasonal or tidal fluctuation.

Prior to sample collection, the characteristics of the surface body will be recorded in the field log book. Size, depth, flow, and location of the sample point in the impoundment or river will be recorded in the field log book. In sampling streams, rivers, and channels sampling will proceed from downstream locations to upstream locations so that sample disturbance will not affect

sampling quality. Also, if both sediment and water samples are being collected at the same time the water samples will be collected first.

2.0 EQUIPMENT

The sampling of sediment in a lake, pond, lagoon, impoundments, streams, rivers, and channels will be achieved by using the following samplers:

1. Trowel or scoop;
2. Sampling trier;
3. Bucket auger;
4. Soil coring device;
5. Veihmeyer sampler;
6. Split spoon sampler; and
7. Ponar dredge.

The factors that will contribute to the selection of a sampler of the stream bed or impoundment include width, depth, flow, and the bottom characteristics of the stream bed or impoundment and whether the sample will be collected from an off-shore or on-shore location.

In collecting sediment samples, care will be taken to minimize disturbance to the sample and to avoid the loss of liquid and fines associated with the sample. The samplers will not decant off any excess liquid. Any liquid that is collected in the bottle is representative of the sediment conditions.

2.1 GENERAL PROCEDURE

The following sampling procedure will be used in the collection of sediment from the shore or bank:

1. Under dry and/or low flow conditions and if the sampling point is within reach, sediment will be collected with a decontaminated trowel or scoop;

2. Once the sample is obtained, it will be transfered directly into the sample bottle provided by the laboratory;

3. Decontaminate the sampling device following the procedures outlined in SOP 6 before collecting the next sample;

4. If the sampling point is under flowing conditions or is greater than 4 inches in depth, a corer or another device that would minimize sample washing will be used;

5. The soil coring device, sampling trier, and the split spoon sampler will allow for the collection of an undisturbed core of sediment. (Follow the procedures outlined in the manufacturers manual for the specific operation of these sampling devices);

6. A decontaminated trowel should be utilized to transfer the sample from the corer directly into a decontaminated sample bottle; and

7. After the collection of the sample, decontaminate the sampling device following the procedures outlined in SOP 6 before collecting the next sample.

WHAT IS TURBIDITY?

Turbidity, cloudiness in water, can be interpreted as an absence of clarity or brilliance. It is caused by suspended and colloidal matter such as clay, silt, organic and inorganic matter and microscopic organisms. Turbidity should not be confused with color since a darkly colored water can still be clear and not turbid.

Turbid water is often an indicator of conditions that could cause damage to manufacturing equipment. Water clarity is especially important to the producers of consumer products such as beverage producers, food processors and water treatment plants. The particulates that cause turbidity may not always be harmful to human health, but are considered an undesirable characteristic.

Turbidity in industrial water used for boiler and cooling systems should be as low as possible. In boilers, the particles may become concentrated and settle out as a sludge that will damage equipment and cause foaming. In cooling water systems, particles can interfere with corrosion inhibitors. Water clarity is improved with fluid-particle separation processes such as sedimentation, coagulation and filtration.

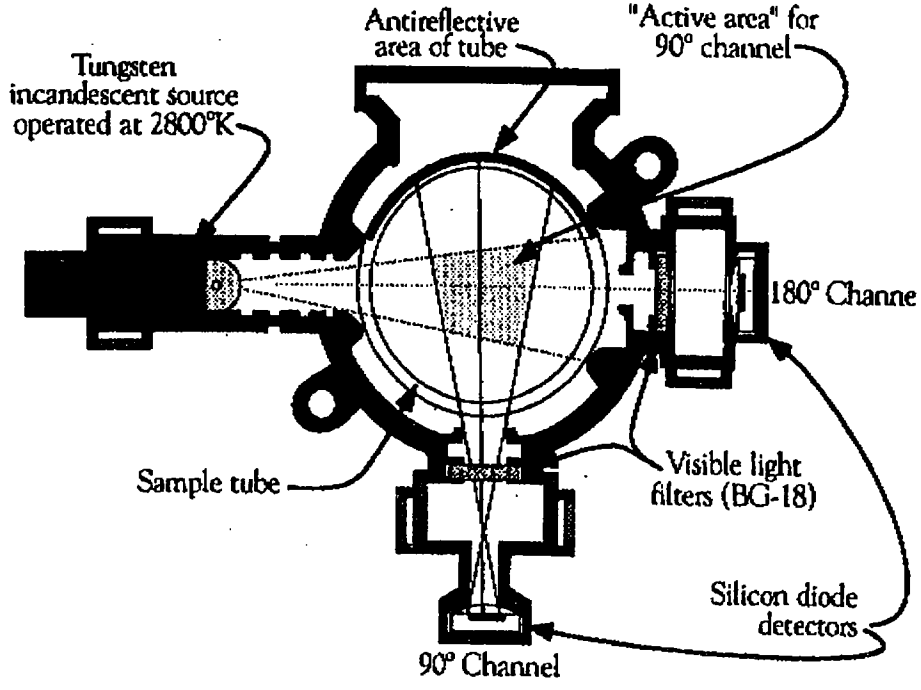
In swimming pools, cloudy water is a common problem. The usual causes for poor water clarity are corrosion, improper filtration and/or improperly balanced water. An algae condition or severe chloramine condition can also cloud pool water.

In natural waters, turbidity is an indicator of quality and productivity and can be used to monitor the health of streams and lakes. Turbid water may indicate runoff from construction, agriculture or other types of pollution. Suspended sediment can carry nutrients and pesticides throughout the water system. Suspended particles near the surface absorb additional heat from sunlight, raising the water temperature and blocking out the light needed by submerged aquatic vegetation and bottom dwelling creatures.

HOW IS TURBIDITY MEASURED?

Light passing through clear water will travel in a straight line. Particles in turbid water will cause the light to scatter giving it a "cloudy" appearance. The turbidity of a sample is determined by measuring the amount of scatter when a light is passed through a sample. The higher the turbidity, the greater the amount of scatter.

Turbidity can be measured in many ways. Visual methods include, the comparative methods, the Secchi disk method and the Jackson Candle method. Comparative methods are used in shallow water and determine turbidity by matching the turbidity of a water sample to a standard of known turbidity either with a "target" at the bottom of a tube or with a turbidity comparator. In the deeper waters of lakes, ponds, rivers and estuaries the Secchi disk is often used to measure turbidity. The Secchi disk is a disk about eight inches in diameter that is either white or is marked with black and white quadrants. The disk is lowered into the water on a calibrated line and the depth is noted where the disk just disappears from sight. The disk is then raised until it is visible. The average of these two distances is known as the "Secchi depth".



2020 Nephelometer

At waterworks and wastewater treatment plants the Jackson Candle apparatus was a standard instrument for measuring turbidities of incoming raw waters and treated wastewater effluents for many years. The equipment was modified over time but originally it consisted of a long glass tube supported over a "standard candle." Water was added to or removed from the tube until the image of the candle flame became indistinct. The depth of the water in the tube was read off a calibrated scale etched into the side of the tube, and results were reported numerically as Jackson Turbidity Units (JTU). The lowest turbidity that can be determined with this method is 25 Nephelometric Turbidity Units (NTU). Since the EPA's Surface Water Treatment requirements state that, finish water from municipal treatment plants will have a turbidity less than 1 NTU, indirect methods were developed to measure turbidity. Turbidimeters are the preferred method.

Nephelometers, such as the 2020, are turbidimeters that measure the scattered light at 90 degrees from the light source. A reference beam passes through the sample and is measured at 180 degrees. The ratio of these two readings is electronically converted to a turbidity measurement in NTU.

GENERAL OPERATING INFORMATION

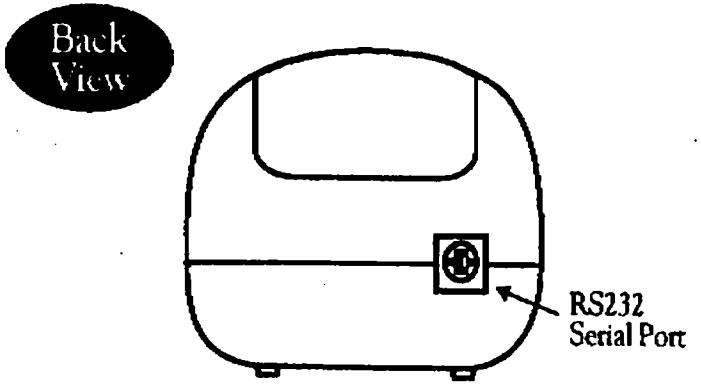
OVERVIEW

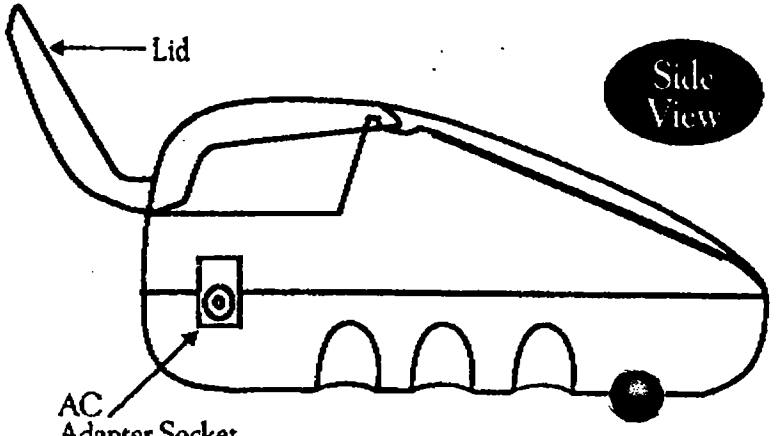
The 2020 Turbidimeter is a portable, microprocessor controlled nephelometer. A multi-detector optical configuration assures long term stability and minimizes stray light and color interferences. All readings are determined by the process of signal averaging over a 5 second period, minimizing fluctuations in readings attributed to large particles and enabling rapid, repeatable measurements. It has a sealed keypad. The microprocessor enables auto-ranging over the full range of 0 to 1100 NTU and provides direct digital readout with a resolution of 0.01 NTU for the lowest range.

The optics feature a tungsten bulb light source with a life expectancy of 800 hours. The light is detected by a silicon photo diode.

The 2020 is supplied with a 9 volt alkaline battery and an AC power adapter.

A RS-232 serial port on the back of the meter allows an interface of the turbidimeter with an IBM compatible computer for real time data acquisition and data storage using the PC. This port also allows an interface with a RS-232 serial printer.





STANDARD SOLUTIONS

The 2020 has been pre-calibrated in the range of 0 to 1100 NTU with AMCO™ primary standards manufactured by Advanced Polymer Systems, Inc. This allows the 2020 to be used for treated water, natural water or wastewater. Recalibration of the 2020 by the user is not required. However, a procedure to standardize the calibration should be performed to obtain the most accurate readings over a narrow range.

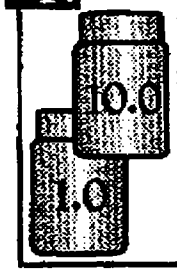
Two AMCO™ standards of 1.00 NTU and 10.0 NTU are supplied with the 2020. Standards of other values are available as accessories. The standards are a suspension of uniformly sized plastic "micro spheres" in ultra pure water, which require no preparation and are stable for long periods of time. These standards were manufactured specifically as a reference to calibrate the 2020. Only LaMotte specific AMCO™ standards should be used with the 2020. These standards are guaranteed to be accurate to within ±1%, if the following precautions are observed:

- The standards will remain stable for up to 4 years prior to opening if stored between 10 and 40°C.
- Once the seal of the bottle is broken, the stability of the standard is only guaranteed for 1 year if stored between 10 and 40°C.
- Never pour any unused or used standard back into the primary standard bottle.
- Do not open the bottle in a dusty or dirty environment. Dust and contaminants from the air can ruin the quality of the standard solutions.
- Before filling a tube with a standard, rinse the inside of the tube with a small amount of standard.
- Cap the standard bottle and the tube immediately after filling.

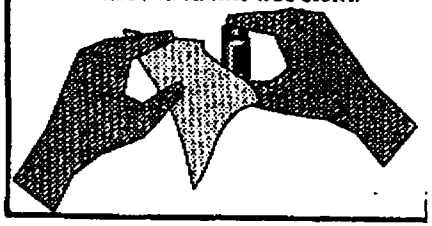
With proper preparation techniques, freshly prepared Formazin standards should be equivalent to the AMCO™ standards and can be used for meter calibration. Correct procedures and approved methods for the use of Formazin standards can be found in the current edition of Standard Methods for Examination of Water and Wastewater.

CALIBRATION PROCEDURE

1. Select a LaMotte AMCO™ 2020 Standard in the range of the samples to be tested. NOTE: Only use LaMotte AMCO™ Standards specific to the 2020 Turbidimeter. Contact LaMotte for replacement standards.

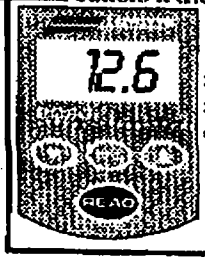


2. Fill a turbidity tube with the standard, cap, and wipe the tube clean with a lint-free cloth.

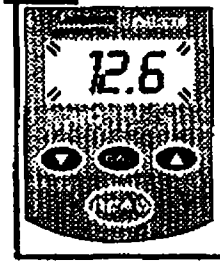


3. Open the lid of the meter. Align the indexing arrow mark on the tube with the indexing arrow mark on the meter, and insert the tube into the chamber.

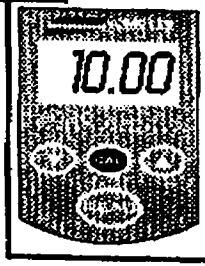
4. Close the lid. Push the **READ** button. If the displayed value is not the same as the value of the reacted standard (within the specification limits), continue with the calibration procedure.



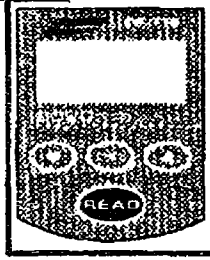
5. Push the **CAL** button for 5 seconds until **CAL** is displayed. Release button. The display will flash. Adjust the display with the ▼ and ▲ buttons until the value of the standard is displayed.



6. Push the **CAL** button again to memorize the calibration. The 2020 display will stop flashing. Calibration is complete.



7. Turn the unit off by holding the **READ** button down for at least 1 second, or proceed to measure the test samples following the procedure on page 19.



Note

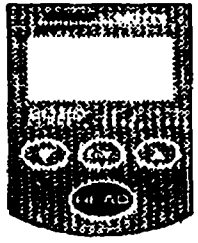
The calibration procedure should be followed once a week, or more often as required by regulations and laws for compliance monitoring. The calibration of the meter is independent of the operating mode.

SELECTING THE EPA MODE

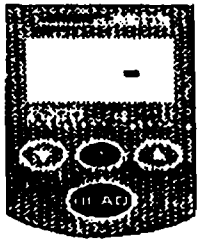
The 2020 turbidity meter has two operating modes, the standard operating mode and the EPA mode. The meter can only be switched from one mode to the other while turning the 2020 on, from the OFF state. The 2020 will remain in which ever mode it was last used, even if the meter has been turned OFF.

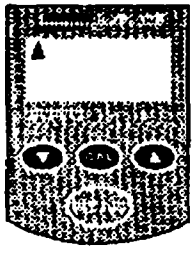
To switch from one mode to the other mode:

1. Turn OFF the 2020, if it is on.



2. Press **CAL** button and hold it down while pressing the **READ** button to turn the meter on.



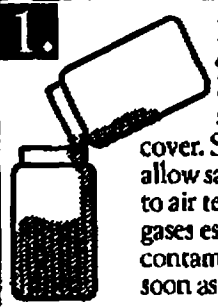


The meter will come on in the opposite mode than it was in previously. (While in EPA mode the ▲ will be visible on the display).

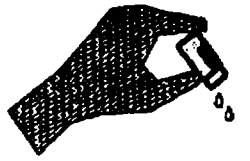
The standard operating mode displays the measured turbidity to the full resolution of the meter. The EPA mode displays the measured turbidity rounded to the reporting requirements of the EPA and Standard Methods compliance monitoring programs. This greatly simplifies the reporting requirements by eliminating the need for the user to manually round off the results according to EPA specifications. The EPA requires these reporting requirements because it recognizes the inherent accuracy of turbidity measurements within the specified ranges.

Note: The calibration of the meter is independent of the operating mode.

1. Fill a clean container with at least 50 mL of sample water and cover. Set sample aside to allow sample to equilibrate to air temperature and let gases escape. Avoid contaminants. Analyze as soon as possible.

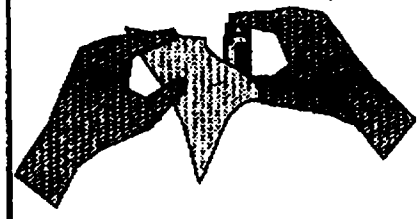


2. Rinse an empty turbidity tube with a portion of the sample. Shake out excess water.

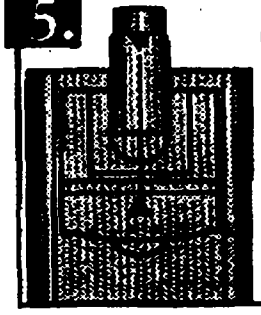


3. Fill the turbidity tube (0286) to the neck by carefully pouring the sample down the side of the tube to avoid creating bubbles.

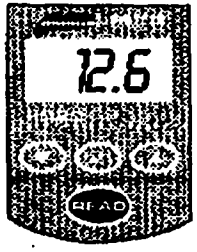
4. Cap the tube and wipe tube dry with a clean lint-free tissue.



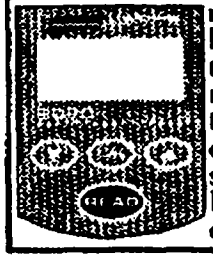
5. Open the 2020 lid. Align the indexing arrow on the tube with the indexing arrow on the meter. Insert the turbidity tube into chamber.



6. Close the lid. Push the **READ** button. The turbidity in NTU units will be displayed within 5 seconds.



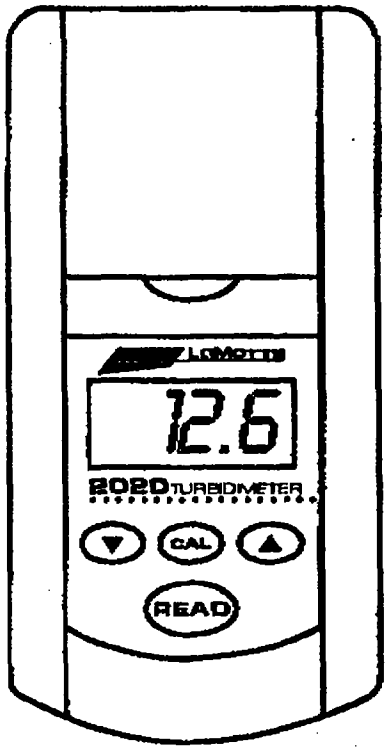
7. The 2020 will turn off automatically 2 minutes after the last button push. To turn the meter OFF manually, hold the **READ** button down for at least 1 second. Release the button when OFF is displayed.



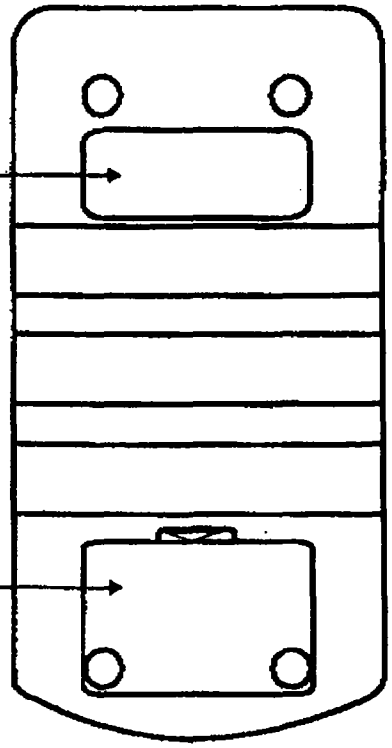
Note

If the sample is higher than 1100 NTU, it must be diluted and retested. See pages 20-22.

Top View



Bottom View

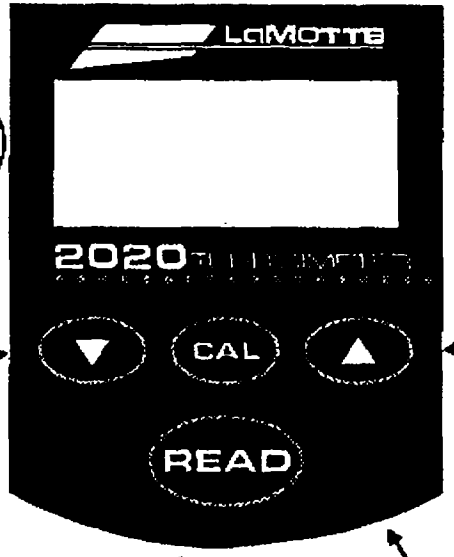


THE KEYPAD

The DISPLAY will display turbidity reading with the following resolution:
0.00 - 10.99 NTU; 11.0 - 109.9 NTU; 110 - 1100 NTU

- When the **READ** button is first pushed, a number will be briefly displayed that indicates the software version number.
- A walking dash "-" will be displayed when measurement is taking place.
- The display will flash after the **CAL** button has been pushed during the calibration procedure until the **CAL** button has been pushed again to enter the adjusted value.
- "*OFF*" will be displayed after the **READ** button has been held down for 1 second. The meter will turn off when the button is released.
- "*Er1*" will be displayed when the battery voltage is very low.
- "*Er2*" will be displayed when measured turbidity is over range (1100 NTU).
- "*Er3*" will be displayed when the bulb has burned out or the tube is misaligned.
- "BAT" will be displayed when the battery voltage is getting low. Readings are reliable. Replace battery as soon as possible.
- "▲" will be displayed when the meter is in EPA mode.

See TROUBLE SHOOTING GUIDE page 23



The DOWN ARROW will **DECREASE** the numerical value of the display while in calibration mode.

The UP ARROW will **INCREASE** the numerical value of the display while in calibration mode.

The READ button is used to turn the meter ON and to take readings. Pressing the button for 1 second will cause the meter to display OFF. Releasing the button when OFF is displayed turns the meter OFF.

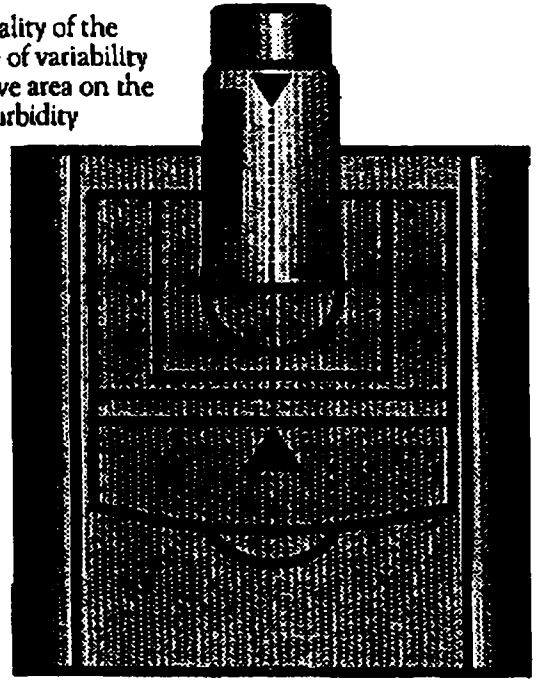
The CAL button is used for **CALIBRATION** procedures and to change between standard operating mode and EPA mode.

TURBIDITY TUBES

Turbidity tubes should always be washed prior to use. Use a mild detergent to remove any dirt or finger prints. Dry the outside of the turbidity tubes with a clean, lint-free cloth or disposable wipe. Allow the turbidity tubes to air-dry in an inverted position to prevent dust from entering the tube.

The handling of the turbidity tubes is of utmost importance. Scratches, fingerprints and water droplets on the turbidity tube or inside the light chamber can cause stray light interference leading to inaccurate results. It is imperative that the turbidity tubes and light chamber be clean and dry. Scratches and abrasions will permanently affect the accuracy of the readings. Tubes can be acid washed periodically and coated with special silicon oil to mask imperfections in the glass. After a tube has been filled and capped, it should be held by the cap and the outside surface should be wiped with a clean, lint-free absorbent cloth until it is dry and smudge-free. Handling the tube only by the cap will avoid problems from fingerprints. Always set the clean tube aside on a clean surface that will not contaminate the tube.

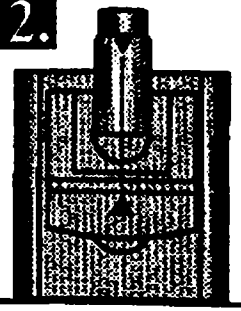
Variability in the geometry and quality of the glassware is the predominate cause of variability in results. The special anti-reflective area on the 2020 tubes allows more accurate turbidity readings for low NTU samples. Only 2020 tubes should be used with the 2020 turbidimeter. Orientation of the tube in the chamber will greatly affect the test results. To obtain the most accurate results, the tubes must be positioned so that the arrow-shaped index mark on the tube is aligned with the arrow-shaped index mark molded into the housing in front of the light chamber. This will ensure that the most accurate results are obtained.



The 2020 turbidity tubes are optically selected but very small variations in the tubes may cause different readings on the same sample in low turbidity water. If greater accuracy is required, such as for Drinking Water requirements, the tubes supplied with the 2020 should be individually calibrated. This procedure is important for reading below 10 NTU but is probably not needed for samples above 10 NTU.

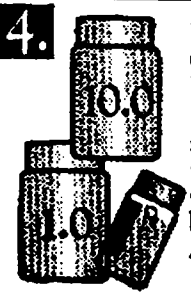
1. Fill each tube (0286) with high-quality water. (Generally distilled water or drinking water is sufficient.)

2. Record a turbidity reading for each tube following the instructions for turbidity measurement on page 21.

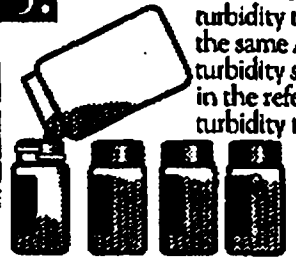


3. Mark the tube with the lowest reading with an "R", for reference turbidity tube, near the top of the tube with a permanent marker.

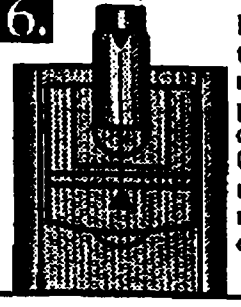
4. Follow the Calibration Procedure on page 16 using the reference turbidity tube, turbidity-free water, and the 1.00 NTU or 10.0 NTU AMCO™ standard.



5. Fill the remaining turbidity tubes with the same AMCO™ turbidity standard as in the reference turbidity tube.



6. Insert each tube into the meter with the proper orientation, Close lid and record the reading for each tube.



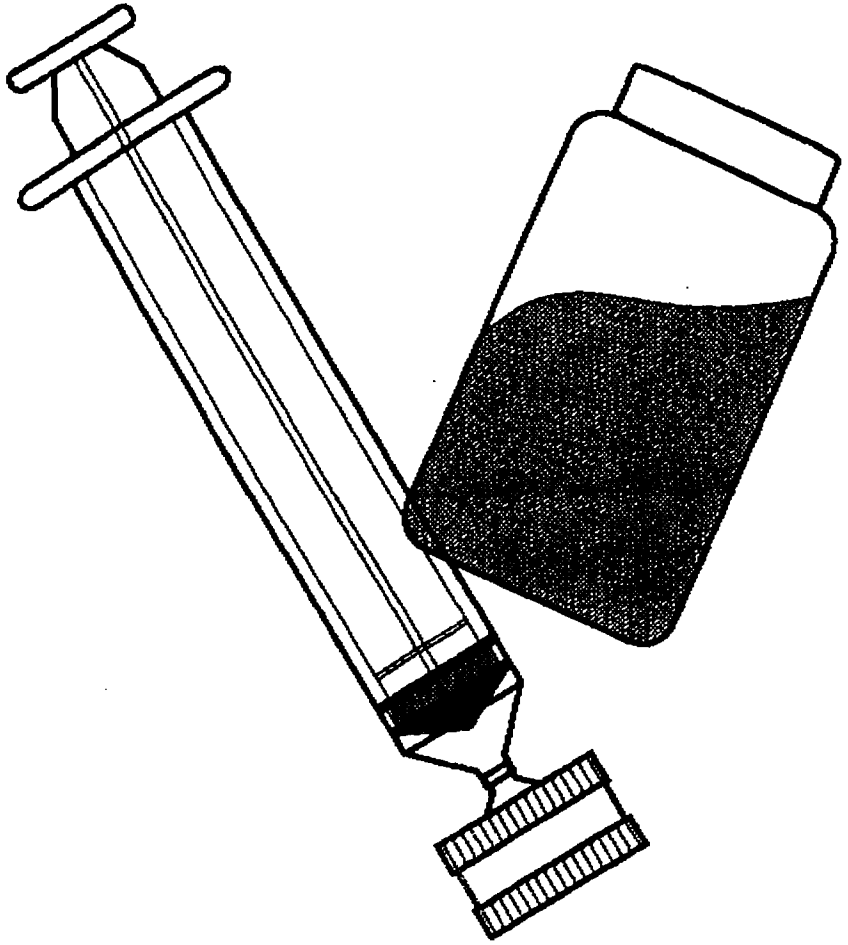
7. The difference between the theoretical value (1.00 NTU or 10.0 NTU) and the actual value is the correction factor for that tube. This factor should be used when comparing results from different tubes.

PREPARATION OF TURBIDITY FREE WATER

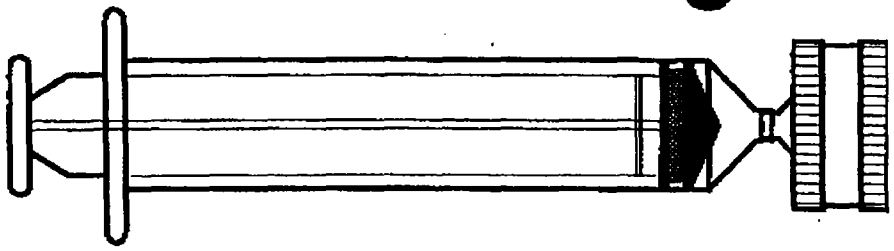
An accessory package (Code 1800, not included) is available for preparing turbidity free water for dilution of high turbidity samples.

The preparation of turbidity free water requires careful technique. Introduction of any foreign matter will affect the turbidity reading. A filtering device with a special membrane filter is used to prepare turbidity-free water. The filter, filter holder, and syringe must be conditioned by forcing at least two syringes full of deionized water through the filtering apparatus to remove foreign matter. The first and second rinses should be discarded. Turbidity-free water as prepared below may be stored in the dark at room temperature in a clean glass bottle with a screw cap and used as required. The storage container should be rinsed thoroughly with filtered deionized water before filling. The water should be periodically inspected for foreign matter in bright light.

See procedure on next page...



PROCEDURE:



1. Unscrew the top of the filter holder (0598). (The black O-Ring should remain in the top part of the filter holder). Place a white membrane filter (1103) on the screen inside. Position the filter disk so that it covers the entire surface of the screen. Replace the top of the filter holder and screw on securely. Note: the membrane filters are white and packaged between two blue protective disks. Handle membrane filters with extreme care.

2. Remove the plunger from the syringe (0943). Attach filter holder to the bottom of the syringe.

3. Pour approximately 50 mL of deionized water into the barrel and exert pressure on the plunger to slowly force the water through the filter. Collect water in a clean container.

4. Remove the filter holder from the syringe, then remove the plunger from the barrel. (This step is required to prevent rupturing the membrane filter by the vacuum as the plunger is removed.)

5. Replace the filter holder and repeat steps 3 and 4 until the desired amount of turbidity-free water has been collected.

Periodically examine the membrane filter to insure that no holes or cracks are present. Depending on the nature of the unfiltered water, it is possible to prepare a liter or more of turbidity-free water using a single filter. The membrane filter may be stored in the holder indefinitely and used as required.

DILUTION PROCEDURES

If a sample is encountered that is higher than 1100 NTU, a careful dilution will bring the sample into the acceptable range. However, there is no guarantee that halving the concentration will exactly halve the NTU values. The particulates often react in an unpredictable manner when diluted.

TESTING TIPS

1. Samples should be collected in a clean glass or polyethylene container.
2. Samples should be analyzed as soon as possible after collection.
3. Discard tubes that are badly scratched.
4. Gently mix sample by inverting before taking a reading but avoid introducing air bubbles.
5. Turbidity readings will be affected by electric fields around motors.
6. Carbon in the sample will absorb light and cause low readings.
7. Observe shelf life recommendations for turbidity standards.
8. The turbidimeter should be placed on a surface free from vibration. Vibrations can cause high readings.
9. Excessive color in a sample will absorb light and cause high readings. The user should verify if a certain level of color will cause a significant error at the level of turbidity being tested.

TROUBLESHOOTING

PROBLEM	CHECK	ACTION
Meter won't turn on	Battery	Replace
	AC Adapter	Plug in
	AC Wall Outlet	Verify power source
	Contact LaMotte for Return Authorization	Return to LaMotte for repair
Suspect Calibration	Check calibration with standards	Use new standards
	Verify standards with Formazin	Run alternate test with Formazin
	Verify with another meter	Check other meter calibrations
	Check tube alignment	Re-align tube
	Check sample tubes for dirt and scratches	Check, clean and/or replace if necessary
	Check to see if internal meter components are wet	Always dry tubes before inserting. Examine chamber for visible moisture.
	Contact LaMotte for Return Authorization	Return for calibration check
Er1	Very low battery	Change battery
Er2	Over range	Dilute sample
Er3	Burnt out bulb or misaligned tube	Check tube alignment Call LaMotte
BAT	Low battery	Change battery

RS232 PORT

The 2020 Turbidimeter may be interfaced with any IBM compatible computer using an Interface cable (Code 1772). The meter may also be interfaced with an RS-232 serial printer, using an appropriate cable and setting the printer configuration to the output below.

Output: RS232 compatible, asynchronous serial, 9600 baud, no parity, 8 data bits, 1 stop bit.

Computer Connection: RS232-(1772) interface connection, 8 pin mDIN/9 pin F D-submin.

Pin out:

5	RS-232 TxD
3	RS-232 RxD
4, 6, 8	digital ground

MAINTENANCE

REPLACING THE BATTERY

The LaMotte 2020 uses a standard 9-volt alkaline battery that is available worldwide. The battery compartment is located on the bottom of the case. To replace the battery:

1. Open the battery compartment lid
2. Remove the battery and disconnect the battery from the polarized plug.
3. Carefully connect the new battery to the polarized plug and insert it into the compartment.
4. Close the battery compartment lid

REPLACING THE LAMP

The tungsten lamp included with the model 2020 has a life of approximately 800 hours. If the display becomes unstable when using LaMotte AMCO™ standards, call LaMotte Company for a return authorization number to have the lamp replaced and have the unit examined.

REPAIRS

If it is necessary to return the instrument for repair, telephone LaMotte Company at 1-800-344-3100 or fax 1-410-778-6394 for a return authorization number.

5. Ensure the proper operation of equipment prior to leaving for the site (this includes having fully charged batteries).

2.2 OPERATION

2.2.1 Calibration

1. With the meter off, adjust the zero adjustment screw located on the face of the meter so the needle indicates zero.

2. Turn the instrument switch to BATT+, then BATT-, to see if the needle is in the battery OK range. If not, replace batteries.

3. To calibrate the conductivity meter, use a conductivity solution equal to 1,000 umhos/cm.

4. The conductivity probe must be rinsed with distilled water before and after every measurement.

5. Turn the instrument on and connect the conductivity probe. Turn RANGE knob to x10 and put the probe in the calibration solution and adjust the conductivity scale to 1,000 umhos/cm by adjusting the STD knob.

6. If the calibration procedures are ineffective, carefully clean the probe with distilled water and soap and repeat the procedure. If the calibration is still ineffective, internal calibration may be necessary and should be adjusted only by the manufacturer.

2.2.2 Data Collection

1. Turn the RANGE switch to the highest range position (1,000). Rinse the probe with distilled water.

2. Insert the probe into the unknown solution at least one inch without touching the sides or bottom of the container.

3. Decrease the range one step at a time until the meter reading is between 10% and 90% of full scale.

4. It may take several minutes for the reading to stabilize when the temperature of the solution is different from the test environment. The probe will automatically compensate for sample temperatures between 5 degrees Celsius and 45 degrees Celsius.

5. After determining the measurement range, chose a standard within that range and calibrate the meter.

6. Re-measure the unknown solution.

7. Record the sample number and location, date, time, operator, and results in the field logbook.

2.2.3 Data Reduction and Interpretation

There is no data reduction required for this instrument. Because the instrument measures an indicator parameter, interpretation of data are straight forward.

2.3 POST OPERATION

2.3.1 Field

If the equipment has come in contact with potential contaminants, decontaminate all equipment as described in Procedure 6. If the equipment has become dirty, be sure to clean off visible dust or dirt. Always clean the probe with distilled water and wrap with a damp paper towel immediately after each use. Damage can occur to the electrode if an acidic, alkaline, or organic rich solution is left on the electrode.

Prior to leaving the site each day the following items will be completed:

1. Record all observations and notes in the logbook.

2. Complete all logbook entries which will include project name and number, date, field time, personnel, visitors on-site, weather conditions, site conditions and any other pertinent data.

3. Review logbook entries and verify accuracy of entries. Sign the last page of that day's entry.

2.3.2 Office

All water quality instruments will be cleaned in the laboratory by the FEM before the instrument is used again. When returning the instrument and supplies to the FEM, the following items will be completed:

1. Report any equipment malfunction and/or damage.

2. Copy field logbook for project file.

3. Copy any field maps used for project file.

REFERENCE SOURCES

Operating Manual for Conductivity Meter, Cole Palmer

Cole Palmer
7425 North Oak Park Ave.
Chicago, IL 60648
Telephone (312) 647-7600

INSTRUCTION MANUAL

CONDUCTIVITY METER

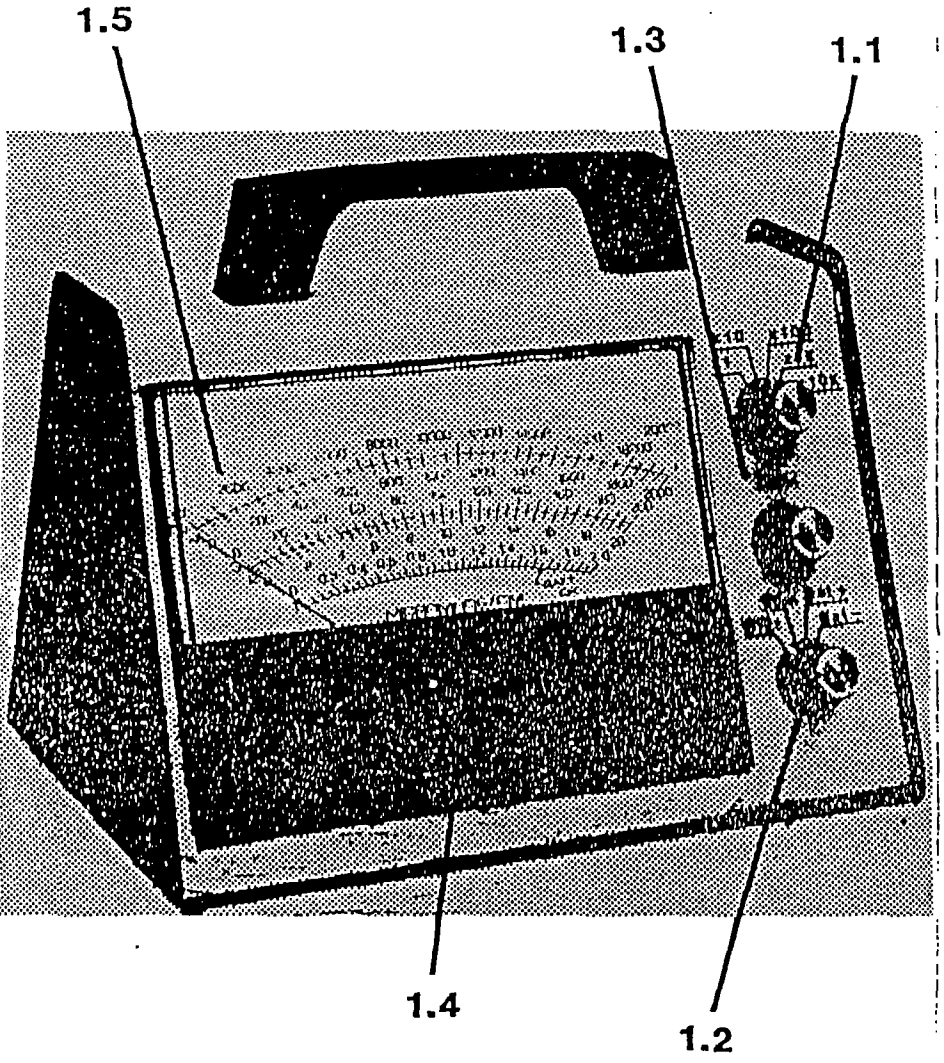
QUICK OPERATION

1. OPEN SHIPPING BOX - TAKE OUT INSTRUMENT
2. ANY DAMAGE?
   -REPORT TO FREIGHT COMPANY
   -CALL DISTRIBUTOR
3. CONNECT CONDUCTIVITY PROBE
4. TURN INSTRUMENT ON (CHECK BATT +, BATT -)
   NOTE: BATTERIES ARE INCLUDED!
5. TURN RANGE SWITCH TO POSITION x1k
6. CENTER CAL KNOB
7. RINSE PROBE IN D.I. WATER
8. INSERT PROBE IN SAMPLE
9. DECREASE RANGE SWITCH UNTIL READING IS BETWEEN 10% AND 90% OF SCALE
10. CHOOSE CALIBRATION STANDARD FOR THAT RANGE
11. RINSE PROBE IN D.I. WATER
12. MEASURE CALIBRATION STANDARD, AND ADJUST CAL KNOB FOR CORRECT READING
13. RINSE PROBE IN D.I. WATER
14. MEASURE SAMPLE

-----READ INSTRUCTION MANUAL-----

CONDUCTIVITY METER

TABLE OF CONTENTS



1.0 INSTRUMENT FAMILIARITY
(FRONT PANEL)

1.1 **FUNCTION: Off/On/Batt+/Batt-**
Off: Turns power off.
On: Activates meter.
Batt+: Displays pos. battery voltage.
Batt-: Displays neg. battery voltage.

1.2 **RANGE:** Five position switch used to select the proper conductivity scale.

x.1.....0-2 micromhos
x 1.....0-20 micromhos
x 10.....0-200 micromhos
x 100.....0-2000 micromhos
x 1k.....0-20,000 micromhos

1.3 **CAL:** Adjustment used to standardize meter with conductivity standards.

1.4 **ZERO ADJUSTMENT SCREW:** Zeros analog meter.

1.5 **ANALOG METER:** 0-20 micromho scale. Multiplier on range switch allows for scale conversion. Batt ok scale indicates region where battery voltage is adequate for operation. A reading below the scale indicates batteries need to be replaced.

1.5a **DIGITAL METER:** Digital readout of conductivity and battery voltages. Batt Lo will appear when batteries reach approximately 4.0 volts.

(REAR PANEL)

1.6 **PROBE INPUT:** 5 pin din connector for detachable conductivity probe.

1.7 **POWER:** Input for optional wall plug adapter.

6.0 THEORY

6.1 Conductivity is the measurement of the amount of electrical current that will flow across two noble metal surfaces when a constant voltage is applied. Conductivity is a nonselective measurement with any charged ion contributing to the total conductivity. Organic compounds such as phenols, alcohols, oils, etc., do not dissociate (ionize) in water and therefore have little or no effect on the conductivity. Conductivity is normally expressed as micromhos per centimeter. In the International System of Units (SI) conductivity is expressed as millisiemens per meter, where 1 mS/m is equal to 10 micromhos per centimeter or 1 S/cm is equal 1 micromhos per centimeter.

CONDUCTIVITIES OF SOME COMMON LIQUIDS

Freshly distilled water	.5 to 2.0 micromhos/cm
Potable water	50 to 1500 micromhos/cm
Normal saline	18,400 micromhos/cm

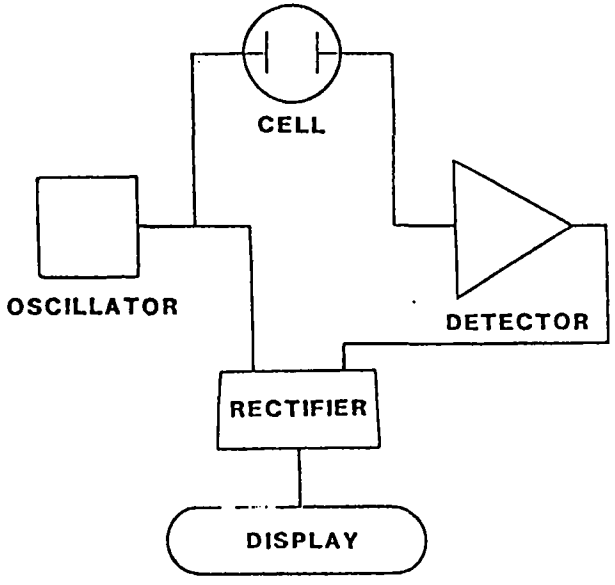
6.2 Conductivity can be used to determine concentration. A nearly linear relationship exists between conductivity and ion concentration for most ionic compounds below 2,000 micromhos/cm. As a result conductivity is often measured to determine total dissolved solids (TDS). It is important to note that this is only a valid methodology when the ionic solution is composed of a pure compound since the exact relationship between conductivity and concentration varies with each ionic compound.

Some examples of the relationship between concentration and conductivity are:

SALT	*CONCENTRATION*	*CONDUCTIVITY* (25 degrees C)
Calcium Carbonate (CaCo)	1 G/L (1000 ppm)	2300 micromhos/cm
Sodium Chloride (NaCl)	1 G/L (1000 ppm)	1990 micromhos/cm
Potassium Chloride (KCl)	1 G/L (1000 ppm)	1880 micromhos/cm

7.0 CIRCUIT FUNCTION

An AC voltage is applied to the conductivity cell. The signal passed to the detector is proportional to the resistance and capacitance of the cell and the sample. The synchronous rectifier, which is controlled by the oscillator, corrects for the capacitive effect. A purely conductive reading is displayed.



1. Lab - a. Unscrew (4) feet, remove shroud.
   b. Loose screws on back panel.
   c. Disconnect battery snaps.
   d. Pull battery packs from holder.
   e. Replace all batteries, noting polarity.
   f. Reassemble instrument.

2. Field - a. Remove battery packs from foam insert, disconnect snaps.
   b. Replace all batteries, noting polarities.
   c. Reassemble instrument.

B. If battery checks are ok and meter fails to respond, have instrument serviced.

11.0 SPECIFICATIONS

	71100	71150	71200	71250
READOUT	6" ANALOG METER	3 1/2 DIGIT LCD	6" ANALOG METER	3 1/2 DIGIT LCD
RANGE	0 - 20,000 micromhos			
STEPS	0 - 2, 0 - 20, 0 - 200, 0 - 2000, 0 - 20,000 micromhos			
ACCURACY	+/- 2% Full Scale			
TEMP. COMP.	Automatic 5 - 45 Degrees Celsius			
PROBE	Dip style (platinum plated) not included		Dip style (platinum plated) included	
SIZE	5" H, 8" W, 5" D		4" H, 12" W, 8" D	
WEIGHT	2.7 lbs. (1.2 Kg)		3.9 lbs. (1.8 Kg)	
POWER	8-1.5 V AA Batt. (std.) 110/220 VAC Adaptor (opt.)		8-1.5 V AA Batteries	

REVISED NOVEMBER 6, 1991

PROCEDURE NO. 9

GROUND WATER SAMPLING

REVISED NOVEMBER 6, 1991

PROCEDURE NO. 9

GROUND WATER SAMPLING

1.0 INTRODUCTION

Collecting ground water samples from wells requires precise procedures to ensure that valid and representative data are generated. The procedures will vary only in terms of the equipment used, with other modifications necessary to address well construction and analytical parameters.

2.0 EQUIPMENT

The equipment used for each sampling event will be dependent on the following general areas:

1. Well construction details; and

2. Analytical parameters.

The equipment used for ground water sampling programs will be capable of purging wells and obtaining samples. In some instances the same piece of equipment (e.g. bailers) can be used for both procedures. However, the use of well purging equipment (e.g.

2. Sample containers
3. Preservatives
4. Ice packs
5. Coolers
6. Personal safety equipment
7. Field and travel blanks
8. Field logbooks
9. Sample labels
10. Chain-of-Custody seals
11. Chain-of-Custody forms
12. Sample analysis request forms

3.0 PROCEDURES

All equipment that enters a well will be properly decontaminated prior to and after each use. Moreover, the equipment will be constructed of relatively inert materials which will not leach or absorb contaminants.

3.1 WELL PURGING

Prior to removing any ground water from a well, the electronic water level indicator will be used to obtain a static water level from each well following Procedure 5.

Once the static water level has been obtained, the volume of standing water in the well will be calculated based on the well diameter and depth. The volume can be calculated with the following formula:

$$V = \pi r^2 h \times 7.48 \text{ gal/ft}^3$$

Where:

V = well volume (gallons)

r = inside well radius (feet)

h = length of water column in well

Alternatively, the following representative volumes for various well diameters can be multiplied directly by the length of the water column.

Inside Casing Diameter	Gallons/Linear Foot
2 inches	.16
4 inches	.65
6 inches	1.47
8 inches	2.61

After the well volume is calculated, a minimum of three well volumes will be removed from the well. The only exception to the minimum purging volume is wells that penetrate water zones that

have such limited hydraulic capacity that removing three well volumes is impractical.

Periodic measurements (e.g. every fifth bailer) of pH, temperature and specific conductance will be made as water is purged from the well. The stabilization in these parameters indicates representative formation water has entered the well. If those parameters exhibit significant variability after three well volumes have been removed, additional water will be removed until the parameters stabilize.

Purge water from each well will be placed in a calibrated container in order to determine when three well volumes has been removed. If a pump is used for purging, and a steady flow rate can be maintained, the purged volume can be calculated by timing after the pumping rate is determined. The pumping rate for each well will be determined by evaluating the data collected during the packer tests, rising and/or falling head permeability tests and pump tests.

The selection of well purging equipment will generally be dictated by well diameter, depth, and accessibility. Wells that are 4 inches or larger in diameter, and greater than 25 feet in depth, will be purged with a submersible pump.

3.2 WELL SAMPLING

The sampling device, a dedicated teflon bailer, will be precleaned prior to obtaining the sample. The bailer line (e.g. polypropylene) and disposable gloves will be discarded after sampling each well. Care will be exercised so the bailer line does not contact the ground surface during sampling. The bailer will be slowly lowered into the well to prevent excessive ground water aqitation and aeration. Water in the bailer will be poured directly into the sample containers. Ground water samples analyzed for dissolved metals will be field filtered prior to placement in the appropriate container. The appropriate preservatives will be added to the containers after they are filled or added at the laboratory before the containers are sent to the field. The samples will be maintained at 4°C. It will be noted that most pumps are not suitable for sampling due their effects upon samples for chemical analyses.

3.3 FIELD FILTERING

Ground water samples collected for dissolved metals analysis will be field filtered upon sample collection and prior to preservation. Filtration will be performed using disposible .45 micron filters. Particularly turbid samples may be pre-filtered utilizing a glass fiber filter prior to the .45 micron filter to speed the process.

After filtration, the ground water samples will be placed in laboratory supplied containers, preserved with nitric acid to a pH of less than two, placed on ice, and shipped to the contracted laboratory.

3.4 DECONTAMINATION

All sampling equipment, with the exception of disposable bailer lines will be decontaminated following Procedure 6 between each sampling event. Dedicated check valve bottom fill bailers will be used for each well, to allow the bailers to be decontaminated in a controlled environment (e.g. laboratory) after each sampling round is completed.

Submersible pumps, hoses and electric lines will be wiped and then placed into a container with clean water and laboratory detergent. The water and detergent will be circulated through the pump and discharge hose by pumping. A final equipment rinse and circulation will be made with potable water.

Other types of pumps may require disposal of the discharge hose after each well is purged. The pump body, valves, etc. will then be cleaned following Procedure 6.

ATTACHMENT C

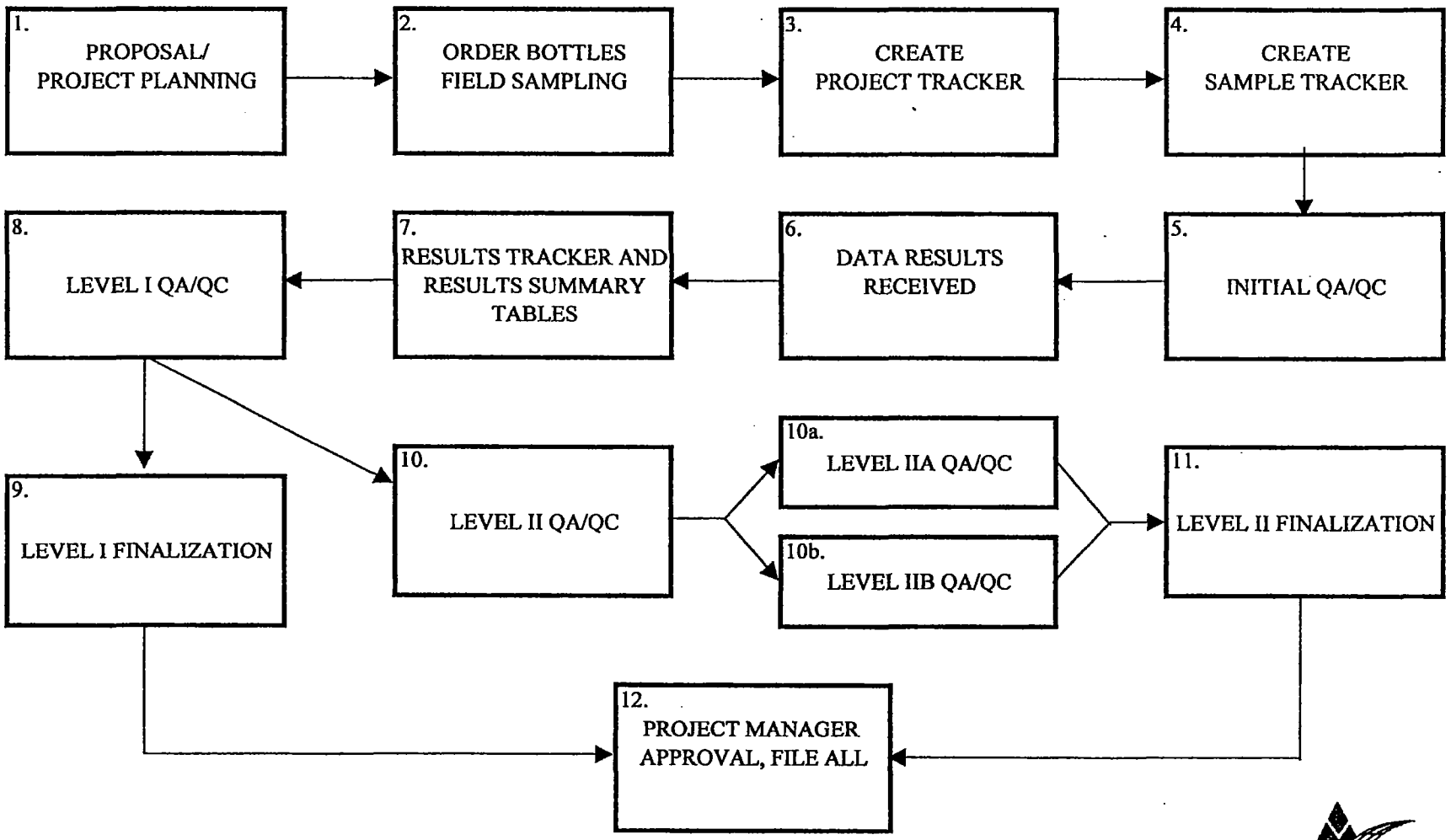
OF THE

QUALITY ASSURANCE PROJECT PLAN

REFINED METALS CORPORATION SITE

DATA MANAGEMENT PLAN

AGC ANALYTICAL DATA MANAGEMENT
STANDARD OPERATING PROCEDURES



Level I QA/QC is the minimim data review required for each project.

#. Identifies the Standard Operating Procedure step. Descriptions of each step are found on attached summary.

☐ Indicates a level of QA/QC review.

AGC ANALYTICAL DATA MANAGEMENT
STANDARD OPERATING PROCEDURE

1. PROPOSAL/PROJECT PLANNING

Initiate sample collection process with Data Validation Specialist. Determine Data Quality Objectives, sampling locations, parameters, analytical methods, data deliverables and schedule.

2. CREATE PROJECT TRACKER

Add project to Project Tracker (see attached example). Project Tracker will contain project name, number and manager, sample location/ID, sample start and completion date, date results expected, level of QA/QC required and date QA/QC completed. A weekly updated printout of the project tracker will be maintained in the QA area for anyone's review.

3. LABORATORY COORDINATION - ORDER BOTTLES, FIELD SAMPLING

Define methodologies to be used, parameters to be analyzed and specific handling/preservation requirements. Order bottles and set schedule with laboratory. Collect samples.

4. CREATE SAMPLE TRACKER

Create sample tracker unless not required for Level I QA/QC (required for Level II QA/QC). The sample tracker will be completed using the chain-of-custody and will include all of the samples collected for a given project. At a minimum, the sample tracker will consist of the AGC location/sample ID, laboratory ID, sample matrix, sample collection date, laboratory performing analysis, analytical parameters and methods, and sample-specific notes. An additional tracker can be created for coordinates if provided. An example of a sample tracker is attached.

5. INITIAL QA/QC

Check chains-of-custodies against the project specifications. Complete an initial QA/QC review (see attached checklist) and file in QA project file. Check that the list of parameters is accurate and note any deviations from sampling plan. Verify that samples have been collected from all locations specified and note deviations from sampling plan. Verify receipt of samples at analytical lab were in good condition and inform project manager if major changes are evident.

6 DATA RESULTS RECEIVED

Upon data receipt, inform project managers unvalidated results are available. Data will be stored in a project file in the QA area with a slip sheet (see attached example) attached describing the review status of the data.

7. RESULTS TRACKER/RESULTS SUMMARY TABLE

Using laboratory diskette deliverables, import data into results tracker. If diskette deliverables are not supplied, enter results from laboratory data package Form Is. Construct data summary tables by exporting the results from the database. The results tracker is

required for Level IIA and Level IIB and data summary tables are required for Level IIB. At a minimum, summary tables will include project name and number, dates of sample collection, sample location/sample ID, parameters analyzed, results, qualifiers, detection limits, qualifier definitions, and signature of QA Scientist. Data summary tables are incomplete until data validation has been completed and all appropriate qualifiers have been added to the data summary tables.

8. LEVEL I QA/QC

Complete Level I QA/QC review checklist (see attached checklist). Verify all analyses were performed as requested and as per method requested (including detection limits). Verify all samples have reported results for the parameters requested and that there are no extreme results. Verify holding times have not been exceeded. Check field blank contamination, field duplicate precision, total versus dissolved results, and sample data (results and forms) where applicable. If any of the preceding do not comply with QA/QC specifications, request additional information or additional analyses from the laboratory as needed and make necessary qualifications.

9. LEVEL I FINALIZATION

Enter required qualifications into results tracker. If Level I QA/QC is the final QA/QC check required then verify that the Level I QA/QC review checklist is complete, update Project Tracker and print out Project Status Form. Copy Level I QA/QC checklist and Project Status Form for project manager to confirm that QA review is complete. Skip to #12.

10. LEVEL II QA/QC

Level IIA or Level IIB Review will be performed.

a. **Level IIA -**

Complete Level IIA review checklist. Review uses QA/QC summary sheets supplied by the analytical laboratory. Validation consists of a review of these summary sheets for laboratory blank contamination, matrix spike and matrix spike duplicate recovery, laboratory and field duplicate relative percent difference, QC check sample recovery, and any additional summary forms (calibration, internal standard areas, etc.). Data results are not recalculated or verified. Any out of criteria QC checks are noted and appropriate data validation qualifiers are applied.

b. **Level IIB -**

Review consists of a full data validation of a "CLP" or "CLP-like" data package. Validation will include examination of all items listed in a Level IIA validation, in addition to all instrument logs, analyst run logs, and chromatographs. All results will be recalculated and verified. A data validation report will be completed describing the usability of the sample data and appropriate data validation qualifiers will be added to the data summary tables.

11. LEVEL II FINALIZATION

To finalized data management, a second QA reviewer will verify consistency between reports, checklists, summary tables and databases and perform data and qualifier checks.

The project tracker, sample tracker, validation reports, validation spreadsheets and sample results database are finalized. The completed validation report and summary tables are copied for the Project Manager for final review.

12. PROJECT MANAGER APPROVAL, FILE ALL

The Project Manager's comments are addressed and, if necessary, data validation reports and summary tables are edited. All data management checklists, tables, and reports are filed in the project file. Data tables for reports can be completed at this time if requested by the Project Manager.

AGC Data Management Project Status Form

Project Manager: SWK PLEASE USE 3 INITIALS

Project Name:

Project Number: 92-002-MP

Event Name: 1STQTR96

Lab: ENSA

Expected Sampling Start Date: 1/1/96

Expected Sampling Complete Date: 3/31/96

Date Results Expected in the Office: 4/30/96

Brief Description of the Event: AIR MONITORING (please be brief)

Notes from the Project Manager:
(to be recorded with the project)

Notes from Data Management:

Level I QA/QC is required for all field sampling events.

Project initiation is required by the Data Management SOP for all projects that require field sampling events. One form is required for each event that will take place in 1996 or 1997.

TO BE COMPLETED BY DATA MANAGEMENT PERSONNEL ONLY

Level I: Y

Level IIA: Y

Level IIB: -

Sample Tracker: Y

Database Checked: Y

Data Review Complete: 6/12/96

Data Package has been forwarded to the Project Secretary for filing.

Data Acceptable as Reported: YES

Data Accepted with Appropriate Qualifiers: N/A

Notes:

- = Not required by the project at this time
X = Required by the project but not complete at this time
Y = Required by the project and complete at this time

EVENT ID	LAB	LAB ID	SAMPLE LOC	COCID	DUPL	MATRIX	DATE SAMPLED	DESCRIPTION
MONTHLY SAMPLING	LLI	273811	AIR STRIPPER EFFLUENT	AIR STRIPPER EFFLUENT		WATER	7/3/97	GRAB SAMPLE
MONTHLY SAMPLING	LLI	273811	TRIP BLANK	TRIP BLANK	TB	WATER	7/1/97	TRIP BLANK
MONTHLY SAMPLING	LLI	276422	AIR STRIPPER EFFLUENT	AIR STRIPPER EFFLUENT		WATER	8/19/97	GRAB SAMPLE
QUARTERLY SAMPLING	LLI		AIR STRIPPER INFLUENT	AIR STRIPPER INFLUENT		WATER	9/4/97	GRAB SAMPLE
MONTHLY SAMPLING	LLI		AIR STRIPPER EFFLUENT	AIR STRIPPER EFFLUENT		WATER	9/4/97	GRAB SAMPLE
QUARTERLY SAMPLING	LLI	277827	AIR STRIPPER INFLUENT	AIR STRIPPER INFLUENT		WATER	9/11/97	GRAB SAMPLE
MONTHLY SAMPLING	LLI	277827	AIR STRIPPER EFFLUENT	AIR STRIPPER EFFLUENT		WATER	9/11/97	GRAB SAMPLE
MONTHLY SAMPLING	LLI	279828	AIR STRIPPER EFFLUENT	AIR STRIPPER EFFLUENT		WATER	10/10/97	GRAB SAMPLE
MONTHLY SAMPLING	LLI	281583	AIR STRIPPER EFFLUENT	AIR STRIPPER EFFLUENT		WATER	11/6/97	GRAB SAMPLE
QUARTERLY SAMPLING	LLI	283677	AIR STRIPPER INFLUENT	INFLU		WATER	12/5/97	GRAB SAMPLE
MONTHLY SAMPLING	LLI	283677	AIR STRIPPER EFFLUENT	EFF		WATER	12/5/97	GRAB SAMPLE
SEMI ANNUAL SAMPLING	LLI	283677	MW-2	MW-2		WATER	12/5/97	GRAB SAMPLE
SEMI ANNUAL SAMPLING	LLI	283677	MW-1	MW-1		WATER	12/5/97	GRAB SAMPLE
SEMI ANNUAL SAMPLING	LLI	283677	MW-7	MW-7		WATER	12/5/97	GRAB SAMPLE
MONTHLY SAMPLING	LLI	285300	AIR STRIPPER EFFLUENT	AIR STRIPPER EFFLUENT		WATER	1/8/98	GRAB SAMPLE
ANNUAL SAMPLING	LLI	285300	VAPOR PHASE CARBON	VAPOR PHASE CARBON		SOLID	1/8/98	COMPOSITE
MONTHLY SAMPLING	LLI	287155	AIR STRIPPER EFFLUENT	AIR STRIPPER EFFLUENT		WATER	2/6/98	GRAB SAMPLE
ANNUAL SAMPLING	LLI	288780	MW-2	MW-2		WATER	3/3/98	GRAB SAMPLE
ANNUAL SAMPLING	LLI	288780	MW-3	MW-3		WATER	3/3/98	GRAB SAMPLE
ANNUAL SAMPLING	LLI	288780	MW-6	MW-6		WATER	3/3/98	GRAB SAMPLE
ANNUAL SAMPLING	LLI	288780	MW-5	MW-5		WATER	3/4/98	GRAB SAMPLE
ANNUAL SAMPLING	LLI	288780	MW-4	MW-4		WATER	3/4/98	GRAB SAMPLE
ANNUAL SAMPLING	LLI	288780	BW-1	BW-1		WATER	3/4/98	GRAB SAMPLE
ANNUAL SAMPLING	LLI	288780	MW-7D	MW-7D	FD	WATER	3/4/98	GRAB SAMPLE
ANNUAL SAMPLING	LLI	288780	MW-1	MW-1		WATER	3/5/98	GRAB SAMPLE
ANNUAL SAMPLING	LLI	288781	AIR STRIPPER INFLUENT	AIR STRIPPER INFLUENT		WATER	3/5/98	GRAB SAMPLE
ANNUAL SAMPLING	LLI	288781	AIR STRIPPER EFFLUENT	AIR STRIPPER EFFLUENT		WATER	3/5/98	GRAB SAMPLE
ANNUAL SAMPLING	LLI	288781	EQUIPMENT BLANK	EQUIPMENT BLANK	EB	WATER	3/3/98	GRAB SAMPLE
ANNUAL SAMPLING	LLI	288781	TRIP BLANK	TRIP BLANK	TB	WATER	3/3/98	TRIP BLANK
ANNUAL SAMPLING	LLI	288780	MW-7	MW-7		WATER	3/4/98	GRAB SAMPLE
FRENCH CREEK	LLI	289862	FRCR-3	FRCR-3		SURFACE WATER	3/24/98	GRAB SAMPLE
FRENCH CREEK	LLI	289862	FRCR-4	FRCR-4		SURFACE WATER	3/24/98	GRAB SAMPLE
FRENCH CREEK	LLI	289862	FRCR-4	FRCR-4D	FD	SURFACE WATER	3/24/98	GRAB SAMPLE
FRENCH CREEK	LLI	289862	FRCR-5	FRCR-5		SURFACE WATER	3/24/98	GRAB SAMPLE
FRENCH CREEK	LLI	289862	FRCR-6	FRCR-6		SURFACE WATER	3/24/98	GRAB SAMPLE
FRENCH CREEK	LLI	289862	FRCR-2	FRCR-2		SURFACE WATER	3/24/98	GRAB SAMPLE
FRENCH CREEK	LLI	289862	FRCR-1	FRCR-1		SURFACE WATER	3/24/98	GRAB SAMPLE
FRENCH CREEK	LLI	289862	TRIP BLANK	TRIP BLANK	TB	WATER	3/24/98	TRIP BLANK
MONTHLY SAMPLING	LLI	290914	AIR STRIPPER EFFLUENT	AIR STRIPPER EFFLUENT		WATER	4/10/98	GRAB SAMPLE

ANALYTES	NOTES
BTEX, PCE, TCE, CARBON CCL4, VINYL CHLORIDE, PH, TSS, TOTAL FE, MN	
BTEX, PCE, TCE, CARBON CCL4, VINYL CHLORIDE	
BTEX, PCE, TCE, CARBON CCL4, VINYL CHLORIDE	
BTEX, PCE, TCE, CARBON CCL4, VINYL CHLORIDE, CHLOROFORM, TRANS-1,2-DCE	RESAMPLED 9/11/97
BTEX, PCE, TCE, CARBON CCL4, VINYL CHLORIDE	RESAMPLED 9/11/97
BTEX, PCE, TCE, CARBON CCL4, VINYL CHLORIDE, PH TSS, FE, MN, CHLOROFORM, TRANS-1,2-DCE	
BTEX, PCE, TCE, CARBON CCL4, VINYL CHLORIDE, PH TSS, FE, MN	
BTEX, PCE, TCE, CARBON CCL4, VINYL CHLORIDE, PH TSS, FE, MN	
BTEX, PCE, TCE, CARBON CCL4, VINYL CHLORIDE, PH TSS, FE, MN	
BTEX, PCE, TCE, CARBON CCL4, VINYL CHLORIDE, PH TSS, FE, MN	
BTEX, PCE, TCE, CARBON CCL4, VINYL CHLORIDE, PH TSS, FE, MN	
CHLOROFORM, TCE, CARBON CCL4	
CHLOROFORM, TCE, CARBON CCL4	
CHLOROFORM, TCE, CARBON CCL4	
BTEX, PCE, TCE, CARBON CCL4, VINYL CHLORIDE, PH TSS, FE, MN	
TCLP VOCS	
BTEX, PCE, TCE, CARBON CCL4, VINYL CHLORIDE, PH TSS, FE, MN	
601/602 LIST	
CHLOROFORM, CARBON TETRACHLORIDE, TCE	
CHLOROFORM, CARBON TETRACHLORIDE, TCE	
CHLOROFORM, CARBON TETRACHLORIDE, TCE	
CHLOROFORM, CARBON TETRACHLORIDE, TCE	
CHLOROFORM, CARBON TETRACHLORIDE, TCE	
601/602 LIST	
601/602 LIST	
PH, TSS, MN, FE, VINYL CHLORIDE, 1,1,DCE, TRANS 1,2-DCE, CHLOROFORM, 1,2-DCA, CARBON TETRACHLORID	
PH, TSS, MN, FE, VINYL CHLORIDE, BTEX, CARBON TETRACHLORIDE, TCE, PCE	
601/602 LIST	
601/602 LIST	
601/602 LIST	
VOA 601/602	
VOA 601/602	
VOA 601/602	
VOA 601/602	
VOA 601/602	
VOA 601/602	
VOA 601/602	
VOA 601/602	
BTEX, CARBON CCL4, PCE, TCE, VINYL CHLORIDE, FE, MN, TSS AND PH	

LAB ID	PARAMETER	T OR L	GROUP	RESULT	UNITS	FLAG	Q	DL
2738115	IRON	T	M	0.042	MG/L			0.002
2738115	MANGANESE	T	M		MG/L	U	U	0.0007
2738115	PH	T	WC	8.03	STD. UNITS			0.01
2738115	TOTAL SUSPENDED SOLIDS	T	WC		MG/L	U	U	3.4
2738115	VINYL CHLORIDE	T	V		UG/L	U	U	2
2738115	CARBON TETRACHLORIDE	T	V		UG/L	U	U	1
2738115	TRICHLOROETHENE	T	V		UG/L	U	U	1
2738115	BENZENE	T	V		UG/L	U	U	1
2738115	TETRACHLOROETHENE	T	V		UG/L	U	U	1
2738115	TOLUENE	T	V		UG/L	U	U	2
2738115	ETHYLBENZENE	T	V		UG/L	U	U	2
2738115	XYLENES (TOTAL)	T	V		UG/L	U	U	1
2738116	VINYL CHLORIDE	T	V		UG/L	U	U	5
2738116	CARBON TETRACHLORIDE	T	V		UG/L	U	U	5
2738116	TRICHLOROETHENE	T	V		UG/L	U	U	5
2738116	BENZENE	T	V		UG/L	U	U	5
2738116	TETRACHLOROETHENE	T	V		UG/L	U	U	5
2738116	TOLUENE	T	V		UG/L	U	U	5
2738116	ETHYLBENZENE	T	V		UG/L	U	U	5
2738116	XYLENES (TOTAL)	T	V		UG/L	U	U	5
2764223	IRON	T	M	0.85	MG/L			0.004
2764223	MANGANESE	T	M	0.0122	MG/L			0.0008
2764223	PH	T	WC	8	STD. UNITS		J	0.01
2764223	TOTAL SUSPENDED SOLIDS	T	WC		MG/L	U	U	3.4
2764223	VINYL CHLORIDE	T	V		UG/L	U	U	2
2764223	CARBON TETRACHLORIDE	T	V		UG/L	U	U	1
2764223	TRICHLOROETHENE	T	V		UG/L	U	U	1
2764223	BENZENE	T	V		UG/L	U	U	1
2764223	TETRACHLOROETHENE	T	V		UG/L	U	U	1
2764223	TOLUENE	T	V		UG/L	U	U	2
2764223	ETHYLBENZENE	T	V		UG/L	U	U	2
2764223	XYLENES (TOTAL)	T	V		UG/L	U	U	1
2778278	IRON	T	M	0.816	MG/L			0.004
2778278	MANGANESE	T	M	0.0032	MG/L			0.0008
2778278	PH	T	WC	6.99	STD. UNITS			0.01
2778278	TOTAL SUSPENDED SOLIDS	T	WC		MG/L	U	U	3.4
2778278	VINYL CHLORIDE	T	V		UG/L	U	U	2
2778278	CARBON TETRACHLORIDE	T	V	6	UG/L			1
2778278	TRICHLOROETHENE	T	V	870	UG/L			10
2778278	BENZENE	T	V		UG/L	U	U	1
2778278	TETRACHLOROETHENE	T	V		UG/L	U	U	1
2778278	TOLUENE	T	V		UG/L	U	U	2
2778278	ETHYLBENZENE	T	V		UG/L	U	U	2
2778278	XYLENES (TOTAL)	T	V		UG/L	U	U	1
2778278	CHLOROFORM	T	V	34	UG/L			1
2778278	TRANS-1,2-DICHLOROETHENE	T	V		UG/L	U	U	2
2778279	IRON	T	M	0.036	MG/L			0.004
2778279	MANGANESE	T	M		MG/L	U	U	0.0008
2778279	PH	T	WC	8.09	STD. UNITS			0.01
2778279	TOTAL SUSPENDED SOLIDS	T	WC		MG/L	U	U	3.4
2778279	VINYL CHLORIDE	T	V		UG/L	U	U	2
2778279	CARBON TETRACHLORIDE	T	V		UG/L	U	U	1
2778279	TRICHLOROETHENE	T	V		UG/L	U	U	1
2778279	BENZENE	T	V		UG/L	U	U	1
2778279	TETRACHLOROETHENE	T	V		UG/L	U	U	1
2778279	TOLUENE	T	V		UG/L	U	U	2
2778279	ETHYLBENZENE	T	V		UG/L	U	U	2
2778279	XYLENES (TOTAL)	T	V		UG/L	U	U	1
2798285	IRON	T	M		MG/L	U	U	0.004
2798285	MANGANESE	T	M		MG/L	U	U	0.0008

NOTES	DILUTION	METHOD
	1	EPA METHOD 200.7
	1	EPA METHOD 200.7
	1	EPA METHOD 150.1
	1	EPA METHOD 160.2
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 200.7
	1	EPA METHOD 200.7
	1	EPA METHOD 150.1
	1	EPA METHOD 160.2
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 200.7
	1	EPA METHOD 200.7
	1	EPA METHOD 150.1
	1	EPA METHOD 160.2
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 200.7
	1	EPA METHOD 200.7
	1	EPA METHOD 150.1
	1	EPA METHOD 160.2
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 624
	1	EPA METHOD 200.7
	1	EPA METHOD 200.7

PROJECT STATUS CHECKLIST

Project Name: _____ Sampling Event/Date(s): _____

Project Number: _____ Laboratory Name: _____

Project Manager: _____

		Date	Initials
1	Project tracker initiated		
2	Samples collected and chain-of-custodies received		
3	1st Internal checklist completed		
4	Fax Recieved		
5	Data deliverables received (Expected on _____)		
6	Project Manager informed that data results have been received		
7	Sample tracker initiated		
8	Level I QA/QC validation started		
9	Level I QA/QC validation completed		
10	Level II A data validation started		
11	Level II A data validation completed		
12	Level II B data validation started		
13	Level II B data validation completed		
14	Data QA spreadsheets constructed		
15	Level II data validation reports and/or checklists verified		
16	Level II data validation reports and/or checklists finalized		
17	Level II QA/QC data validation qualifiers entered into results tracker		
18	Database entries verified		
19	Data management finalized (project tracker, sample tracker, QA/QC spreadsheets, data validation reports and checklists)		

INITIAL QA/QC REVIEW CHECKLIST

Project Name: _____ Sampling Event/Date(s): _____

Project Number: _____ Laboratory Name: _____

Project Manager: _____

1 Verify the parameters listed for analyses on the chain-of-custody are consistent with those listed in the Workplan, QA plan, and/or proposal.

Yes ☐ No ☐

Comments/deviations: _____

2 Verify the methods listed on the chain-of-custody are consistent with those listed in the Workplan, QA plan, and/or proposal.

Yes ☐ No ☐

Comments/deviations: _____

3 Verify all sample locations were sampled, the correct number of samples were collected and the locations are consistent with the Workplan, QA plan, and/or proposal.

Yes ☐ No ☐

Comments/deviations: _____

4 Verify the project tracker is updated.

Yes ☐ No ☐

If no, explain: _____

5 Verify a project status form is initiated.

Yes ☐ No ☐

If no, explain: _____

6 Verify the Project Manager has been informed of any deviations noted on this form.

Yes ☐ No ☐

Additional Comments: _____

QA Scientist: _____ Date: _____

LEVEL I QA/QC DATA REVIEW CHECKLIST

Project Name: _____ Sampling Event/Date(s): _____

Project Number: _____ Laboratory Name: _____

Project Manager: _____ Laboratory Case Number: _____

1 Verify Initial QA/QC review is complete.

Yes ☐ No ☐

If no, complete initial QA/QC review before proceeding with Level I QA/QC review.

2 Verify the chain-of-custody is present.

Yes ☐ No ☐

Comments: _____

3 Verify the sample ID on the chain-of-custody matches the laboratory sample ID.

Yes ☐ No ☐

Comments: _____

4 Verify the analyses requested have been performed.

Yes ☐ No ☐

Comments: _____

5 Verify the analyses were performed using the requested methods.

Yes ☐ No ☐

Comments: _____

6 Verify results were reported for all samples collected and all parameters requested.

Yes ☐ No ☐

Comments: _____

7 Verify no extreme results were reported.

Yes ☐ No ☐

Comments: _____

QA Scientist : _____ Date: _____

LEVEL I QA/QC DATA REVIEW CHECKLIST

Project Name: _____ Sampling Event/Date(s): _____

Project Number: _____ Laboratory Name: _____

Project Manager: _____ Laboratory Case Number: _____

8 Verify samples were analyzed within holding time criteria.

Yes ☐ No ☐

If no, assign the appropriate qualifiers to the affected sample results.

Qualifiers applied: _____

9 Verify there is no field, equipment, or trip blank contamination present.

Yes ☐ No ☐ NA ☐

If no, assign the appropriate qualifiers to the affected sample results.

Qualifiers applied: _____

10 Verify a field duplicate sample was collected and reproducibility is within acceptance criteria.

Yes ☐ No ☐ NA ☐

If no, assign the appropriate qualifiers to the affected sample results.

Field duplicate samples: _____
Qualifiers applied: _____

11 Verify that the total metal results are greater than or equal to the dissolved metal results (total chromium greater than hexavalent chromium).

Yes ☐ No ☐ NA ☐

If no, assign the appropriate qualifiers to the affected sample results.

Qualifiers applied: _____

12 Verify Level I QA/QC review is complete.

Yes ☐ No ☐

13 Verify if Level II QA/QC review is required.

No ☐ Level IIA ☐ Level IIB ☐

Additional Comments: _____

QA Scientist : _____ Date: _____

LEVEL IIA QA/QC DATA REVIEW CHECKLIST

Project Name: _____ Sampling Event/Date(s): _____

Project Number: _____ Laboratory Name: _____

Project Manager: _____ Laboratory Case Number: _____

1 Verify Level I QA/QC review was completed.

Yes ☐ No ☐

Comments: _____

2 Verify a method or laboratory blank was analyzed with the samples and that there is no blank contamination present.

Yes ☐ No ☐

If no, assign the appropriate qualifiers to the affected sample results.

Qualifiers applied: _____

3 Verify the appropriate instrument calibration was performed and that the calibration is acceptable.

Yes ☐ No ☐

If no, assign the appropriate qualifiers to the affected sample results.

Qualifiers applied: _____

4 Verify calibration verification standards were analyzed with the samples and that the calibration verification standard results were within acceptance criteria.

Yes ☐ No ☐

If no, assign the appropriate qualifiers to the affected sample results.

Qualifiers applied: _____

5 Verify a laboratory replicate was performed and that the percent difference is within acceptance criteria.

Yes ☐ No ☐

If no, assign the appropriate qualifiers to the affected sample results.

Qualifiers applied: _____

QA Scientist : _____ Date: _____

LEVEL IIA QA/QC DATA REVIEW CHECKLIST

Project Name: _____ Sampling Event/Date(s): _____

Project Number: _____ Laboratory Name: _____

Project Manager: _____ Laboratory Case Number: _____

6 Verify a laboratory matrix spike was performed and the percent recovery is within acceptance criteria.

Yes ☐ No ☐

If no, assign the appropriate qualifiers to the affected sample results.

Qualifiers applied: _____

7 Verify a laboratory control standard was performed and the percent recovery is within acceptance criteria.

Yes ☐ No ☐ NA ☐

If no, assign the appropriate qualifiers to the affected sample results.

Qualifiers applied: _____

8 Verify a serial dilution was performed and the percent difference is within the acceptance criteria.

Yes ☐ No ☐ NA ☐

If no, assign the appropriate qualifiers to the affected sample results.

Qualifiers applied: _____

9 Verify Level IIA QA/QC review is complete.

Yes ☐ No ☐

Additional Comments: _____

QA Scientist : _____ Date: _____

VOLATILE DATA VALIDATION SUMMARY

Site Name: _____ Laboratory: _____

Project Number: _____ Case/Order No.: _____

Sampling Date(s): _____

Compound List: ☐ TCL ☐ Priority Pollutant ☐ Appendix IX ☐ Other _____

Method: ☐ CLP SOW 3/90 ☐ 40 CFR 136 ☐ SW-846 Method _____ ☐ Other _____

The following table indicates the data validation criteria examined, any problems identified, and the QA action applied.

Data Validation Criteria:	Accept	FYI	Qualify	Comments
Holding Times				
GC/MS Tuning				
Initial Calibrations				
Continuing Calibrations				
Blank Analysis Results				
System Monitoring/Surrogate Results				
MS/MSD Results				
Field Duplicate Results				
Internal Standard Areas/RT				
Target Compound Identification				
TIC Identification				
Quantitation/Detection Limits				
System Performance				
Overall Assessment of Data				
Other:				

General Comments: _____

Accept - No qualification required.
FYI - For your information only, no qualification necessary.
Qualify - Qualify as rejected, estimated or biased.
NR - Not Reviewed
NA - Not Applicable

QA Scientist _____

Date _____

SEMIVOLATILE DATA VALIDATION SUMMARY

Site Name: _____ Laboratory: _____

Project Number: _____ Case/Order No.: _____

Sampling Date(s): _____

Compound List: ☐ TCL ☐ Priority Pollutant ☐ Appendix IX ☐ Other _____

Method: ☐ CLP SOW 3/90 ☐ 40 CFR 136 ☐ SW-846 Method _____ ☐ Other _____

The following table indicates the data validation criteria examined, any problems identified, and the QA action applied.

Data Validation Criteria:	accept	FYI	qualify	Comments
Holding Times				
GC/MS Tuning				
Initial Calibrations				
Continuing Calibrations				
Blank Analysis Results				
System Monitoring/Surrogate Results				
MS/MSD Results				
Field Duplicate Results				
Internal Standard Areas/RT				
Target Compound Identification				
TIC Identification				
Quantitation/Detection Limits				
System Performance				
Overall Assessment of Data				
Other:				

General Comments: _____

Accept - No qualification required.
FYI - For your information only, no qualification necessary.
Qualify - Qualify as rejected, estimated or biased.

NA - Not analyzed.
NR - Not reviewed.

QA Scientist _____

Date _____

INORGANIC DATA VALIDATION SUMMARY

Site Name: _____ Laboratory: _____

Project Number: _____ Case /Order No.: _____

Sampling Date(s): _____

Compound List: ☐ TAL ☐ Priority Pollutant ☐ Appendix IX ☐ Other _____

Method: ☐ CLP SOW ILMO4. ☐ 40 CFR 136 ☐ SW-846 Method ☐ Other _____

The following table indicates the data validation criteria examined, any problems identified, and the QA action applied.

Data Validation Criteria:	accept	FYI	qualify	Comments
Holding Times				
Initial Calibrations				
Continuing Calibrations				
CRDL Standards				
Blank Analysis Results				
ICP Interference Check Sample Recoveries				
Duplicate Results				
Field Duplicate Results				
Spike Analysis Recoveries				
Serial Dilution Results				
Laboratory Control Sample Results				
Furnace AA QC Analysis				
Quantitation/Detection Limits				
Overall Assessment of Data				
Other:				

General Comments: _____

Accept - No qualification required.
FYI - For your information only, no qualification necessary.
Qualify - Qualify as rejected, estimated or biased
NA - Not applicable.
NR - Not reviewed.

QA Scientist _____

Date _____

WET CHEMISTRY DATA VALIDATION SUMMARY

Site Name: _____ Laboratory: _____

Project Number: _____ Case /Order No.: _____

Sampling Date(s): _____

Parameter List: _____

Method: _____

The following table indicates the data validation criteria examined, any problems identified, and the QA action applied.

Data Validation Criteria:	accept	FYI	qualify	Comments
Holding Times				
Calibration Curve				
Initial Calibration				
Continuing Calibration				
Laboratory Control Sample Results				
Blank Analysis Results				
Duplicate Analysis Results				
Field Duplicate Analysis Results				
Matrix Spike Analysis Results				
Quantitation/Detection Limits				
Overall Assessment of Data				
Other:				

General Comments: _____

Accept - No qualification required.
FYI - For your information only, no qualification necessary.
Qualify - Qualify as rejected, estimated or biased

NA - Not Applicable
NR - Not Reviewed

QA Scientist _____

Date _____

TriMatrix Laboratories, Inc.

ANALYTICAL REPORT
USEPA CLP FORM 1

Advanced GeoServices Corporation
Proj: Burgess Battery Site

Subm: November 9, 1998 Samples

Submittal Number: 34836- 6
Location:
Contact: Jennifer L. Rice
Phone: (616) 975-4500

CAS No.		C-2-AR	Data Qualifiers C	Q	M	Units
	Lab Sample No:	210856				
7439-92-1	Lead, Total	* 335			P	mg/kg dry
	Sampled by:	Rieger				
	Date Sampled:	11/09/98				
	Time Sampled:	14:20				
	Date Received:	11/10/98				
	Time Received:	07:30				

* See attached Statement of Data Qualifications.

TriMatrix Laboratories, Inc.

ANALYTICAL REPORT
USEPA CLP FORM 1

Advanced GeoServices Corporation
Proj: Burgess Battery Site

Subm: November 9, 1998 Samples

Submittal Number: 34836- 6
Location:
Contact: Jennifer L. Rice
Phone: (616) 975-4500

CAS No.		B-2-BR	Data Qualifiers C	Q	M	Units
	Lab Sample No:	210857				
7439-92-1	Lead, Total	58			P	mg/kg dry
	Sampled by:	Rieger				
	Date Sampled:	11/09/98				
	Time Sampled:	14:45				
	Date Received:	11/10/98				
	Time Received:	07:30				

TriMatrix
Laboratories, Inc.

ANALYTICAL REPORT
USEPA CLP FORM 1

Advanced GeoServices Corporation
Proj: Burgess Battery Site

Subm: November 9, 1998 Samples

Submittal Number: 34836- 6
Location:
Contact: Jennifer L. Rice
Phone: (616) 975-4500

CAS No.		B-1-FR	Data Qualifiers C	Q	M	Units
	Lab Sample No:	210858				
7439-92-1	Lead, Total	69			P	mg/kg dry

Sampled by:	Rieger
Date Sampled:	11/09/98
Time Sampled:	15:00
Date Received:	11/10/98
Time Received:	07:30

ATTACHMENT D

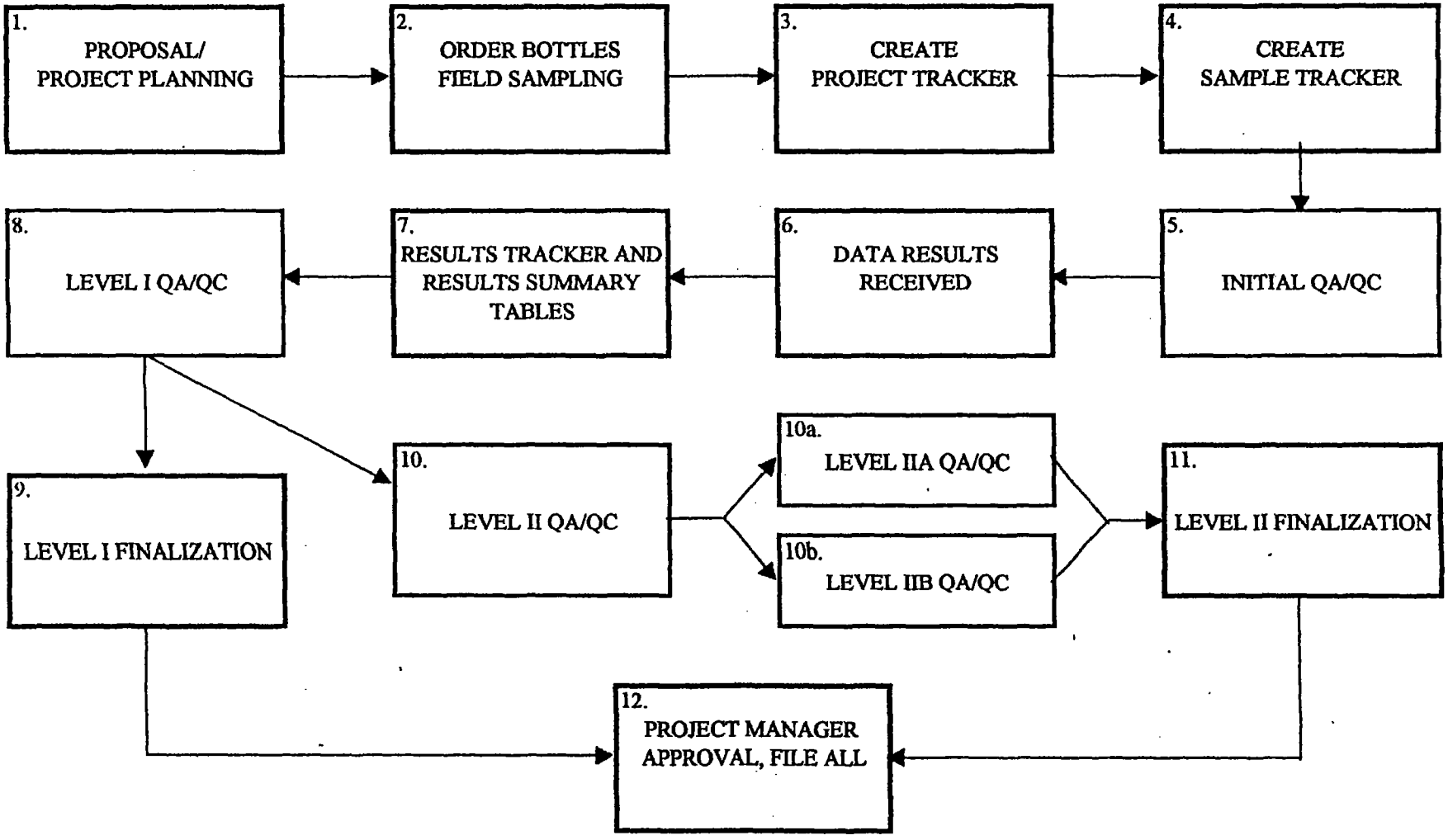
OF THE

QUALITY ASSURANCE PROJECT PLAN

REFINED METALS CORPORATION SITE

DATA VALIDATION CHECKLIST

AGC ANALYTICAL DATA MANAGEMENT
STANDARD OPERATING PROCEDURES



Level I QA/QC is the minimim data review required for each project.

#. Identifies the Standard Operating Procedure step. Descriptions of each step are found on attached summary.

☐ Indicates a level of QA/QC review.

Dvsop

AGC ANALYTICAL DATA MANAGEMENT
STANDARD OPERATING PROCEDURE

1. PROPOSAL/PROJECT PLANNING
   Initiate sample collection process with Data Validation Specialist. Determine Data Quality Objectives, sampling locations, parameters, analytical methods, data deliverables and schedule.

2. ORDER BOTTLES, FIELD SAMPLING
   Order bottles and set schedule with laboratory. Collect samples.

3. CREATE PROJECT TRACKER
   Add project to Project Tracker (see attached example). Project Tracker will contain project name, number and manager, sample location/ID, sample start and completion date, date results expected, level of QA/QC required and date QA/QC completed. A weekly updated printout of the project tracker will be maintained in the QA area for anyone's review.

4. CREATE SAMPLE TRACKER
   Create sample tracker unless not required for Level I QA/QC (required for Level II QA/QC). The sample tracker will be completed using the chain-of-custody and will include all of the samples collected for a given project. The sample tracker consists of the AGC location/sample ID, laboratory ID, sample matrix, sample collection date, laboratory performing analysis, analytical parameters and methods, and sample-specific notes. An additional tracker can be created for coordinates if provided. An example of a sample tracker is attached.

5. INITIAL QA/QC - all chains of custodies checked against the project specifications.
   Complete an initial QA/QC review (see attached checklist) and file in QA project file. Check that the list of parameters is accurate and note any deviations from sampling plan. Verify that samples have been collected from all locations specified and note deviations from sampling plan. Verify receipt of samples at analytical lab were in good condition, and inform project manager if major changes are evident.

6. DATA RESULTS RECEIVED
   Upon data receipt, inform project managers unvalidated results are available. Data will be stored in a project file in the QA area with a slip sheet (see attached example) attached describing the review status of the data.

7. RESULTS TRACKER/RESULTS SUMMARY TABLE
   Using laboratory diskette deliverables, import data into results tracker. If diskette deliverables are not supplied, enter results from laboratory data package Form I's. From the database, export the results to data summary tables. The results tracker is required for Level IIA and Level IIB and data summary tables are required for Level IIB only. At a minimum, summary tables will include project name and number, dates of sample collection, sample location/sample ID, parameters analyzed, results, qualifiers, detection limits, qualifier definitions, and signature of QA Scientist.

8. LEVEL I QA/QC

Complete Level I QA/QC review checklist (see attached checklist). Verify all analyses were performed as requested and as per method requested (including detection limits). Verify all samples have reported results for the parameters requested and that there are no extreme results. Verify holding times have not been exceeded. Check field blank contamination, field duplicate precision, total versus dissolved results, and sample data (results and forms) where applicable. If any of the preceding do not comply with QA/QC specifications, request additional information or additional analyses from the laboratory as needed and make necessary qualifications.

9. LEVEL I FINALIZATION

Enter required qualifications into results tracker. If Level I QA/QC is the final QA/QC check required then verify that Level I QA/QC review checklist is complete, update Project Tracker and print out Project Status Form. Copy Level I QA/QC checklist and Project Status Form for project manager to confirm that QA review is complete. Skip to #12.

10. LEVEL II QA/QC

Level IIA or Level IIB Review will be performed.

a. **Level IIA -**

Review uses QA/QC summary sheets supplied by the analytical laboratory. Validation consists of a review of these summary sheets for laboratory blank contamination, matrix spike and matrix spike duplicate recovery, laboratory and field duplicate relative percent difference, QC check sample recovery, and any additional summary forms (calibration, internal standard areas, etc.). Data results are not recalculated or verified.

b. **Level IIB -**

Review consists of a full data validation of a "CLP" or "CLP-like" data package. Validation will include examination of all items listed in a Level IIA validation, in addition to all instrument logs, analyst run logs, and chromatographs. All results will be recalculated and verified. A data validation report will be completed describing the usability of the sample data.

11. LEVEL II FINALIZATION

To finalized data management, a second QA reviewer will verify consistency between reports, checklists, summary tables and databases and perform data and qualifier checks. The project tracker, sample tracker, validation reports, validation spreadsheets and sample results database are finalized. The completed information is copied for the Project Manager for final review.

12. PROJECT MANAGER APPROVAL, FILE ALL

The Project Manager's comments are addressed and, if necessary, data validation reports and summary tables are edited. All data management checklists, tables, and reports are filed in the project file.

PROJECT STATUS CHECKLIST

Project Name: _____ Sampling Event/Date(s): _____

Project Number: _____ Laboratory Name: _____

Project Manager: _____

		Date	Initials
1	Project tracker initiated		
2	Samples collected and chain-of-custodies received		
3	1st Internal checklist completed		
4	Fax Recieved		
5	Data deliverables received (Expected on _____)		
6	Project Manager informed that data results have been received		
7	Sample tracker initiated		
8	Level I QA/QC validation started		
9	Level I QA/QC validation completed		
10	Level II A data validation started		
11	Level II A data validation completed		
12	Level II B data validation started		
13	Level II B data validation completed		
14	Data QA spreadsheets constructed		
15	Level II data validation reports and/or checklists verified		
16	Level II data validation reports and/or checklists finalized		
17	Level II QA/QC data validation qualifiers entered into results tracker		
18	Database entries verified		
19	Data management finalized (project tracker, sample tracker, QA/QC spreadsheets, data validation reports and checklists)		

INITIAL QA/QC REVIEW CHECKLIST

Project Name: _____ Sampling Event/Date(s): _____

Project Number: _____ Laboratory Name: _____

Project Manager: _____

1 Verify the parameters listed for analyses on the chain-of-custody are consistent with those listed in the Workplan, QA plan, and/or proposal.

Yes ☐ No ☐

Comments/deviations: _____

2 Verify the methods listed on the chain-of-custody are consistent with those listed in the Workplan, QA plan, and/or proposal.

Yes ☐ No ☐

Comments/deviations: _____

3 Verify all sample locations were sampled, the correct number of samples were collected and the locations are consistent with the Workplan, QA plan, and/or proposal.

Yes ☐ No ☐

Comments/deviations: _____

4 Verify the project tracker is updated.

Yes ☐ No ☐

If no, explain: _____

5 Verify a project status form is initiated.

Yes ☐ No ☐

If no, explain: _____

6 Verify the Project Manager has been informed of any deviations noted on this form.

Yes ☐ No ☐

Additional Comments: _____

QA Scientist : _____ Date: _____

LEVEL I QA/QC DATA REVIEW CHECKLIST

Project Name: _____ Sampling Event/Date(s): _____

Project Number: _____ Laboratory Name: _____

Project Manager: _____ Laboratory Case Number: _____

1 Verify Initial QA/QC review is complete.

Yes ☐ No ☐

If no, complete initial QA/QC review before proceeding with Level I QA/QC review.

2 Verify the chain-of-custody is present.

Yes ☐ No ☐

Comments: _____

3 Verify the sample ID on the chain-of-custody matches the laboratory sample ID.

Yes ☐ No ☐

Comments: _____

4 Verify the analyses requested have been performed.

Yes ☐ No ☐

Comments: _____

5 Verify the analyses were performed using the requested methods.

Yes ☐ No ☐

Comments: _____

6 Verify results were reported for all samples collected and all parameters requested.

Yes ☐ No ☐

Comments: _____

7 Verify no extreme results were reported.

Yes ☐ No ☐

Comments: _____

QA Scientist : _____ Date: _____

LEVEL I QA/QC DATA REVIEW CHECKLIST

Project Name: _____ Sampling Event/Date(s): _____

Project Number: _____ Laboratory Name: _____

Project Manager: _____ Laboratory Case Number: _____

8 Verify samples were analyzed within holding time criteria.

Yes ☐ No ☐

If no, assign the appropriate qualifiers to the affected sample results.

Qualifiers applied: _____

9 Verify there is no field, equipment, or trip blank contamination present.

Yes ☐ No ☐ NA ☐

If no, assign the appropriate qualifiers to the affected sample results.

Qualifiers applied: _____

10 Verify a field duplicate sample was collected and reproducibility is within acceptance criteria.

Yes ☐ No ☐ NA ☐

If no, assign the appropriate qualifiers to the affected sample results.

Field duplicate samples: _____

Qualifiers applied: _____

11 Verify that the total metal results are greater than or equal to the dissolved metal results.

Yes ☐ No ☐ NA ☐

If no, assign the appropriate qualifiers to the affected sample results.

Qualifiers applied: _____

12 Verify Level I QA/QC review is complete.

Yes ☐ No ☐

13 Verify if Level II QA/QC review is required.

No ☐ Level IIA ☐ Level IIB ☐

Additional Comments: _____

QA Scientist : _____ Date: _____

INORGANIC DATA VALIDATION SUMMARY

Site Name: _____ Laboratory: _____

Project Number: _____ Case /Order No.: _____

Sampling Date(s): _____

Compound List: ☐ TAL ☐ Priority Pollutant ☐ Appendix IX ☐ Other _____

Method: ☐ CLP SOW ILMO4. ☐ 40 CFR 136 ☐ SW-846 Method ☐ Other _____

The following table indicates the data validation criteria examined, any problems identified, and the QA action applied.

Data Validation Criteria:	accept	FYI	qualify	Comments
Holding Times				
Initial Calibrations				
Continuing Calibrations				
CRDL Standards				
Blank Analysis Results				
ICP Interference Check Sample Recoveries				
Duplicate Results				
Field Duplicate Results				
Spike Analysis Recoveries				
Serial Dilution Results				
Laboratory Control Sample Results				
Furnace AA QC Analysis				
Quantitation/Detection Limits				
Overall Assessment of Data				
Other:				

General Comments: _____

Accept - No qualification required.
FYI - For your information only, no qualification necessary.
Qualify - Qualify as rejected, estimated or biased
NA - Not applicable.
NR - Not reviewed.

QA Scientist _____

Date _____

ATTACHMENT E

OF THE

QUALITY ASSURANCE PROJECT PLAN

REFINED METALS CORPORATION SITE

FIELD AUDIT CHECKLIST

ADVANCED GEOSERVICES CORP.
FIELD OPERATIONS AUDIT CHECKLIST

Project/Site Name: _____ Project Number: _____

Date: _____ Audit Conducted from _____ hour to _____ hour

Audit Team: _____ _____

On-Site Field Personnel: _____ _____

Audit conducted on the following:

_____ Soil Sampling _____ Groundwater Sampling _____ Health & Safety

_____ Surface Water/Sediment _____ Decontamination _____ Other

Brief description of site activities:

_____
_____
_____
_____

Field Logbook Review	Yes	No	N/A
Is the field logbook permanently bound?	_____	_____	_____
Is the field logbook numbered?	_____	_____	_____
Are the pages sequentially number, none missing?	_____	_____	_____
Are all entries made in ink?	_____	_____	_____
Is the end of each entry dated and signed?	_____	_____	_____
Are all blank spaces within the field notes and at the bottom of the page crossed out?	_____	_____	_____

Field Logbook Review (cont.)	Yes	No	N/A
Full name of personnel on-site and their responsibilities noted?	_____	_____	_____
Documentation of agency and client oversight?	_____	_____	_____
Weather conditions noted?	_____	_____	_____
Purpose of operations/description of event included?	_____	_____	_____
Date, time, and details of conversations between team members, client contacts, or other parties documented?	_____	_____	_____
Documentation of approval of any on-site decisions, especially deviations from the approved workplan or QAPjP included?	_____	_____	_____
Field instrumentation used, date and time of calibrations, and any standards used noted?	_____	_____	_____
Field measurement results and person conducting test included?	_____	_____	_____
Date, time, and location of sampling event	_____	_____	_____
Method of sample collection	_____	_____	_____
Sample times, description, identification	_____	_____	_____
Sample Parameters and preservation	_____	_____	_____

Sampling (General)	Yes	No	N/A
Was a copy of the QAPjP available on-site?	_____	_____	_____
Field checklist on hand and followed?	_____	_____	_____
Is a site specific map indicating sampling locations available?	_____	_____	_____

Sampling (General) (cont.)	Yes	No	N/A
Were the sampling locations the same as those specified in the sampling plan?	_____	_____	_____
If no, were proper procedures used to obtain approval of the change?	_____	_____	_____
Was the new location documented in the field logs with reason for change?	_____	_____	_____
Were samples collected as specified in the QAPjP?	_____	_____	_____
Were the appropriate bottles used for sample collection (correct type and size)?	_____	_____	_____
Were sample bottles labeled properly?	_____	_____	_____
Were samples properly preserved?	_____	_____	_____
Were volatile samples collected first?	_____	_____	_____
Were the proper sample volumes procured?	_____	_____	_____
Were correct decontamination procedures used?	_____	_____	_____
Were samples iced while in field and for shipment?	_____	_____	_____
Did potential for cross-contamination exist?	_____	_____	_____
Was the sampling technique consistent for all samples?	_____	_____	_____
Were samples properly packaged for shipment (packed to avoid breakage, sufficient ice packs, and sufficient absorbent material included)?	_____	_____	_____
Were chain-of-custody documents filled out completely (client, location, date, time, matrix, no. of bottles etc.) and packaged with the samples?	_____	_____	_____
Were custody seals initialed and placed on bottles?	_____	_____	_____

Sampling (General) (cont.)	Yes	No	N/A
Was strapping tape used on the coolers and were the coolers adequately labeled?	_____	_____	_____
Was sample custody maintained after sample collection?	_____	_____	_____

Sampling (Soils)	Yes	No	N/A
Were the samples collected at the proper depths?	_____	_____	_____
Were the samples screened with an HNU or OVA?	_____	_____	_____
Was a description of the material documented?	_____	_____	_____
Were VOAs collected prior to homogenization?	_____	_____	_____
Were samples homogenized correctly?	_____	_____	_____

Sampling (Surface Water/ Sediments)	Yes	No	N/A
Were the stream flow and velocity noted?	_____	_____	_____
Was sampling performed downstream to upstream?	_____	_____	_____
Were the samplers standing downstream of sample collection location?	_____	_____	_____
Were samples collected in non-stagnant areas?	_____	_____	_____
Were sediments characterized as to type and size?	_____	_____	_____
Were pH, DO, conductivity and temperature taken?	_____	_____	_____

Sampling (Groundwater)	Yes	No	N/A
Were bailer and bail line dedicated to each well?	_____	_____	_____

Sampling (Groundwater)cont.	Yes	No	N/A
Were the purge water and pump lines decontaminated or disposed properly?	_____	_____	_____
Were well volumes and purge rates properly calculated and documented?	_____	_____	_____

Field Quality Control Samples	Yes	No	N/A
Were field duplicates collected at the proper frequency?	_____	_____	_____
Were field blanks collected?	_____	_____	_____
Were equipment blanks collected for each matrix and all appropriate equipment?	_____	_____	_____
Was the proper water source used for field and equipment blank collection?	_____	_____	_____
Were trip blanks included in the bottle shipment?	_____	_____	_____

Field Measurements	Yes	No	N/A
Was all field equipment calibrated properly and at the frequency required?	_____	_____	_____
Were calibration times and procedures documented in the field logbook?	_____	_____	_____
Were the proper standards used?	_____	_____	_____

Decontamination	Yes	No	N/A
Was the proper decontamination method performed?	_____	_____	_____
Was equipment decontaminated at the proper frequency?	_____	_____	_____

Decontamination (cont.)	Yes	No	N/A
Was equipment dedicated to each sampling location?	_____	_____	_____
Was the proper decontamination area used?	_____	_____	_____

Health and Safety	Yes	No	N/A
Was a health and safety briefing conducted and noted in the logbook prior to start of work?	_____	_____	_____
Was the proper level of personal protection used and documented?	_____	_____	_____
Was the HASP readily available?	_____	_____	_____
Were emergency contacts readily available?	_____	_____	_____
Was monitoring equipment present?	_____	_____	_____
Was a first aid kit readily available ?	_____	_____	_____
Was contaminated clothing disposed of properly?	_____	_____	_____

Overall Comments	Yes	No	N/A
Were the personnel conducting the investigation professional?	_____	_____	_____
Were the project objectives understood by the personnel?	_____	_____	_____
Was the field crew organized?	_____	_____	_____
Was there continuity in the process?	_____	_____	_____
Did weather conditions affect the sample quality?	_____	_____	_____

Audit Summary and Comments

_____

_____

_____

_____

_____

_____

_____

_____

_____

Signed by: _____ Print: _____

_____ _____

_____ _____

Date: _____

ATTACHMENT F

OF THE

REFINED METALS CORPORATION SITE

TRIMATRIC 1998 AUDIT REPORT

LABORATORY AUDIT REPORT

OF

TRI MATRIX LABORATORIES, INC.

AUDIT DATE: MAY 28,1998

FOR

ORGANIC AND INORGANIC ANALYSES

PREPARED BY:

ADVANCED GEOSERVICES CORP.
CHADDS FORD, PENNSYLVANIA

JUNE 12, 1997

LABORATORY AUDIT
TRIMATRIX LABORATORIES, INC
GRAND RAPIDS, MICHIGAN

1.0 INTRODUCTION

An audit of the TriMatrix Laboratories, Inc. (TriMatrix) facility of Grand Rapids, Michigan was conducted on May 28, 1998 by Advanced GeoServices Corp.(AGC) as required by the Morgan County Quality Assurance Project Plan for Gould Electronics, Inc. The on-site laboratory audit was conducted while samples were being processed and analyzed by the laboratory.

Laboratory audits are conducted to determine whether a laboratory follows proper sample custody and preservation protocols, preforms sample preparation and analyses according to specified methodologies, and implements quality assurance/quality control procedures. Laboratory audits also determine overall laboratory staff qualifications and experience, as well as the facility's sample capacity. Data package deliverables are also audited to identify deficiencies that may be a problem when analytical results are reported.

This audit was conducted on the analytical areas involving organic and inorganic sample preparation and analysis. In addition, administrative, custody, quality control/assurance, data reduction/data package preparation, documentation, and client service areas of direct impact to the laboratory's ability to meet project objectives, good laboratory practices, and EPA documentation and requirements were audited. The standard operating procedures (SOPs) and laboratory quality assurance manual were also reviewed.

2.0 AUDIT ORGANIZATION

2.1 Auditor

Advanced GeoServices Corp. Quality Assurance Scientist:	Denise McGuire

2.2 TriMatrix Laboratories, Inc. Personnel Contacted during Audit

Laboratory Manager	Douglas Kriscunas
Quality Assurance Manager	Rick Wilburn
Project Chemist	Jennifer Rice
Client Service Bottle Preparation	Daniel VanderBorgh
Sample Receipt Officers	Keith Banchoff
	Deborah Sidlauskas
Metals Preparation	Margaret Scott
Metals Furnace Analyst	Denise Coffey
Metals ICP Analyst	David Johnson
Organic Preparation	Scott Hetrick
MS Volatile/Semivolatile Supervisor	Janet Kudirka

Ms. McGuire was accompanied by Rick Wilburn and Jennifer Rice throughout the audit.

3.0 AUDIT SUMMARY

3.1 Laboratory Facility

3.1.1 General

TriMatrix Laboratories, Inc.(TriMatrix), Grand Rapids, Michigan, is one of three full-service environmental laboratories in the TriMatrix laboratory network. TriMatrix (formally Earth Tech laboratories) entered the environmental services over 20 years ago. As a medium sized laboratory, TriMatrix provides analysis for industrial waste management and environmental monitoring programs. The laboratory's services include water, wastewater, soil, sediment, and sludge analyses. Laboratory certifications include State of New Jersey, Michigan, Wisconsin, Minnesota, Arizona, and California.

3.1.2 Laboratory Address and Phone Number

The laboratory location is:

TriMatrix Laboratories, Inc.
5555 Glenwood Hills Parkway, SE
P.O. Box 49588-8692
Grand Rapids, Mi. 49588

Phone and Facsimile numbers are:

(616) 975-4500 (Phone)
(616) 942-7463 (Fax)

3.1.3 Laboratory Layout/Security

The facility, a combination of office and laboratory space, was designed by their own chemists and constructed specifically for the purpose of analyzing environmental samples. The laboratory is comprised of approximately 12,900 square feet building built in 1988. The laboratory is divided into office space, sample storage and receipt areas, bottle preparation area, sample preparation areas and several inorganic and organic laboratories. At the time of the audit plans were in effect to break ground for a new laboratory facility which will provide almost double the current space.

The current laboratory facility has a main entrance, a side entrance for pick-up and delivery of samples and several other entrances and emergency exits. The reception area and sample receiving area are reported to be continually staffed during normal working hours. All other exterior doors are locked. All visitors must be accompanied by laboratory personnel before proceeding into the laboratory.

The doors into the laboratory space and walk-in sample cooler are locked at all times and can only be opened using a combination. The combination for these doors is reported changed several times a year and only given to laboratory personnel.

The laboratory working hours are 8:00 am to 6:00 pm 5 days a week. During working hours, samples are received by sample custodians. Before and after working hours sample drop off can be arranged as often second shift personnel are present in the laboratory.

3.2 Organization and Personnel

The Managing Partners of Envirotech remain actively involved with the every day operations at this single laboratory facility. The Managing Partners are chemists, engineers and businessmen who advised AGC that they act as proactive advisors to current regulators on technical advisory committees, therefore, remaining informed on regulatory affairs. The staff of approximately 75 individuals include chemists, scientists, technicians, computer data management personnel and engineers. Specific personnel education and experience requirements under Contract Laboratory Program (CLP) are required since the laboratory provide EPA CLP testing procedures. The individuals met during the audit and a review of the professional profiles reveals that most personnel possess undergraduate degrees and the personnel meet or exceed the CLP minimum experience requirement. In addition, supervisory and management personnel appeared well experienced with many years of environmental analytical chemistry experience. The laboratory appears adequately staffed, properly experienced, and capable of meeting routine and non-routine project requirements.

3.3 Sample Receipt and Storage Area

Sample custodians are present to prepare and distribute sample bottles and accept sample deliveries 5 days a week. Sample pick up and drop off hours are between 8:00 am and 6:00 pm. Special arrangements can be made for sample pick-up or drop-off outside of these hours. When samples are received, the sample receipt officer checks the sample bottles against the chain-of-custody and checks for custody seals, pH and temperature. The temperature is tested using an infrared gun. All coolers are checked for temperature with up to three readings taken per sample cooler. pH is taken by placing a small amount of sample into a jar and placing pH paper into the sample. The result is compared to the pH scale located on the pH paper box. Volatile organic samples are not checked for pH at log-in, but are instead checked prior to analysis in the volatile organics laboratory. Preservatives are added to the samples which were not properly preserved. Containers are also checked for damage, appropriate volume, container type and preservation. This information is recorded on a sample receiving and log-in checklist. If the custody seal is broken, the bottle is leaking, or any other problem is observed, it is noted on the problem submittal report and given to the project chemist, who then informs the client immediately.

The sample custodian or assistant assigns an unique laboratory ID number and affixes a pre-printed laboratory ID sticker to the sample bottle. The sample numbers and chain-of -custody information are entered into the Laboratory Information Management System (LIMS). The LIMS system has the ability to store all information appropriate to the client and sampling event.

All sample bottles for analyses other than VOA are placed in the walk-in refrigerator. The walk-in refrigerator has a locked opening into the sample receipt area and the metals preparation area. The sample containers requiring VOA analyses are transported to the VOA laboratory, where they are placed in secured, locked refrigerators. The temperature of all storage areas are monitored and recorded daily.

Each chain-of-custody is compared with the LIMS printout by the project chemist. Holding times, analyses requested and any other client specific instructions are verified at this time.

Bottle Preparation Area

The sample bottles are prepared by the sample custodian at the laboratory for distribution. All bottles are purchased from a vendor, precleaned. Preserved bottles are clearly labeled with color coded tags. Deionized water from the volatile laboratory is used for trip blanks and water identical to that used for method blanks is used for field blanks. Since the water used for method blanks and dilutions does not undergone the same osmosis procedures for all analysis, the type of water provided for field blanks is dependent on the type of analysis to be performed. In other words, a large bottle of DI water to be used for all field blank analysis is not provided. Instead, separate containers for specific analyses are provided with method specific water.

For the newly approved methanol extraction method, VOA jars are weighed and labeled with an identification number. The identification number is documented along with the weight. With the VOA jars, plastic ampules with 10 mls of methanol are provided. This eliminates the holding time requirement for methanol preserved VOA jars and the possibility of methanol evaporation. The ampule is broken in the field after soil collection and added to the soil in the sample jar.

Besides the requested sample containers, all sample coolers are equipped with chain-of-custodies, temperature bottles, packing material and plastic bags for ice.

3.4 Sample Preparation Areas

The inorganic and organic preparation areas are separate from the instrumentation laboratories. The inorganic area, however, is at one end of the wet chemistry laboratory. The preparation areas were maintained in a clean and orderly manner with the exception of a couple of unidentifiable sample bottles on the window sill. SOPs are written for each procedure and available for all analysts. Preparation logbooks were kept in a neat and consistent manner. Review of the logbooks is periodically performed by department supervisors.

All primary standards are purchased and traceable. Reference materials/ standards are labeled and dated. Calibration solutions are purchased pre-made, and diluted to required concentrations. All

working solutions and spiking solutions are recorded in a logbook with lot numbers of the primary standard and preparation date recorded. Deionized water is used for method blanks, spiking solution preparation and sample preparation. Glassware is hand washed.

Samples were being prepared for inorganic analyses during the audit. Samples prepared for ICP analyses were not always digested. Most undigested samples were checked for precipitate after adding nitric acid. If precipitate formed, the samples were digested. The laboratory fortified blank was spiked with metal analytes prior to digestion, whereas the laboratory control sample (LCS) was not digested at all.

The Supervisors use the LIMS printout to schedule the analyses to be performed on each day. It is the Supervisors' responsibilities to make certain the analysts perform the appropriate analysis on the sample within holding time. All preparations and extractions take place under exhaust hoods which are constantly monitored for adequate air flow.

Recommendation: It was noticed that unlabeled samples were left on a window sill. It was unapparent why these samples were not disposed of since it was obvious that they were not being used at this time for analysis. Samples need to always be labeled and once an analysis is complete the samples should be disposed of properly.

Recommendation: At present dissolved water samples, drinking waters and some clear samples are not digested prior to metal analysis by ICP. All samples should be digested for ICP analyses, especially when using the Trace ICP. Recent studies have indicated organic compounds present in the undigested samples interfere with the analyses by Trace ICP and may cause false positive results.

Recommendation: LCS samples should be digested prior to analysis.

3.5 Analyses Areas

The instrumentation metals area was well maintained in a clean and orderly manner. Although there was little unoccupied bench space, due to numerous instruments, the analysts appeared to have adequate space. The volatiles laboratory also had little unoccupied bench space, but was orderly and clean. Volatile analyses and metals analyses were being performed at the time of the audit. At present there was only one analyst running the furnace analyses and one analyses running the ICP analyses. Analyst were cross trained on the furnace and one ICP, but only one analyst was trained on the trace ICP.

Instrumentation manuals, SOPs, and EPA methods were readily available for the analysts. Maintenance logs for all instruments are maintained. The analysts follow EPA protocol including the analysis of standards, laboratory control samples, preparation blanks, matrix spikes and matrix spike duplicates. All samples analyzed were documented on run chronicles. All data from one run is complied and reviewed by the chemist. The chemist verifies that all data are reported accurately, the methods are followed correctly, and that all method required quality control samples were performed

and are within the acceptance limits. The data is then given to data entry personnel who enter the results into the LIMS. All data is checked by another data entry personnel who verifies the data entry. If there are any questions with the data, it is returned to the analyst for confirmation. All analysts put together their analytical runs with all the support documentation. The volatile supervisor checks the analysts work prior to submitting the data to the project chemist. It was not apparent that the inorganic data was reviewed by a supervisor. The project chemist is responsible for reviewing the data for completeness and to make certain all project specific items were performed.

All stock solutions were purchased and EPA traceable. The solutions are labeled and dated upon opening. Unlocked and locked refrigerators are maintained in the analyses areas for samples and standards. The refrigerator temperatures are monitored daily and kept in a bound logbooks.

Recommendation: Additional cross training needs to be incorporated. More than one analyst needs to be able to analyses samples on any one instrument.

Recommendation: All data should be reviewed by a supervisor prior to being entered into the LIMS.

3.6 Client Services

Prior to the start of any project, a client services representative is designated as the project's laboratory project chemist. All requests for bottles, changes in the scope of work, inquiries about the data or any other project specific information is directed to the laboratory project chemist. It is the laboratory project chemist's responsibility to document all deviations from the Project Quality Assurance Project Plan and notify the client. The laboratory project chemist will address all data inquiries, contacting the necessary laboratory personnel for additional information, as required, to address the inquiries in the most accurate and efficient way.

3.7 Data Reduction/Data Package Preparation

Analytical results are directly imported from the analytical instruments into the reporting database for most analyses. This minimizes the number of manual transcriptions, thus reducing the potential of reporting errors. The results which are hand entered into the system are verified by another individual. The data are to be reviewed by both the analyst and supervisor prior to being downloaded into the reporting database. The reporting databases are secured by means of a password protection system that accounts for any changes made to the database and by whom. The Document Control Officer assembles the project-specific data package consisting of all analyses, writes the narratives and spot checks for all associated data. Before a data package is released to the client, a Managing Partner reviews the package by performing recalculations and verifying a percentage of the reported results.

Recommendations: The client Service Representative should look over the data packages prior to release as to make certain client specific needs are met.

3.8 Quality Control/Quality Assurance

Quality control manuals and SOPs are available to all personnel. A Managing Partner is the Quality Assurance Officer. The Quality Assurance Officer performs a review following the review of the analyst conducting the test and the department supervisor. Data reliability and validity is ensured by constant documentation, supervisory review, quality assurance review, laboratory quality control samples, and EPA traceable standards. SOPs and the Quality Control manual were being updated when the audit was conducted.

4.0 SUMMARY

The Tri Matrix facility in Grand Rapids, Michigan has a good operating quality assurance/quality control program in place.

The overall review of the laboratory indicated the laboratory personnel to have the expertise, experience, and capability to perform analysis of various environmental matrices including, soil, sediment, water and wastewater. The laboratory has sufficient capacity and equipment to meet Advanced GeoServices analytical efforts. The laboratory is capable of producing USEPA CLP deliverables, QC summary packages and a standard format. Upon request, diskette deliverables will also be provided in a format to meet our needs.

Laboratory data packages are complete and easy to validate. When full deliverables are required, a summary package is provide prior to the complete data package. Evening and weekend shifts are beneficial to quick turn-around projects. The personnel are experienced, up to date with current environmental regulations and available to answer analytical questions. TriMatrix does not have the cross training on the Trace ICP; therefore, fast turn around for metals to be analyzed by Trace ICP must be scheduled in advanced. It should also be noted that at this time total organic halogen (TOX) analyses is sub contracted to an outside laboratory and TriMatrix does not have an Ion Chromatograph (IC) instead nitrite, nitrate and phosphate are analyzed using a Letchet analyzer.

TriMatrix should be considered by AGC for analyses of all media for all environmental analyses for all projects, especially projects in Region V due to their location.